

Bureau of Mines  
Report of Investigations 4796



OIL-SHALE OPERATIONS IN NEW SOUTH WALES,  
AUSTRALIA

BY A. J. KRAEMER AND H. M. THORNE

United States Department of the Interior— July 1951

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\* \* \* \* \* **Report of Investigations 4796**



UNITED STATES DEPARTMENT OF THE INTERIOR  
Oscar L. Chapman, Secretary  
BUREAU OF MINES  
James Boyd, Director

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Work on manuscript completed April 1951. The Bureau of Mines will welcome reprinting of this paper, provided the following footnote acknowledgment is made: "Reprinted from Bureau of Mines Report of Investigations 4796."

July 1951



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A. J. Kraemer<sup>1/</sup> and H. M. Thorne<sup>2/</sup>

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## INTRODUCTION

The following account of oil-shale operations in Australia is based upon information obtained by the authors while visiting Australia from September 15 to October 7, 1947, supplemented by published data, and augmented by analyses of samples of oil shale and its products at the Oil-Shale Research and Development Laboratories of the Bureau of Mines at Laramie, Wyo.

The primary objective of the authors' trip to Australia was to obtain, first hand, information regarding the equipment and methods used by National Oil Proprietary, Ltd., at Glen Davis, New South Wales, in mining oil shale, extracting oil from it, and refining the shale oil. This company was operating the only oil-shale plant that had been active in Australia since 1945. The plant comprised an oil-shale mine, 104 modified Pumpherson (Fell) retorts in 2 batteries, a shale-oil refinery, and a 3-inch gasoline pipeline to the railroad loading terminal at Newnes Junction, 30 miles away. There were, also, the usual and necessary auxiliary facilities, such as a power house, mechanical shops, offices, and laboratories.

Information regarding the company's operations was wanted because of its bearing upon the synthetic liquid fuels program of the Bureau of Mines, pursuant to the Synthetic Liquid Fuels Act of April 5, 1944 (30 U.S.C., 321-325, as amended). One objective of the Bureau's program, in accordance with the mandate of the Act, is to furnish American industry cost and engineering data necessary for the development of a synthetic-liquid fuel industry, and an obvious means of obtaining some of these data is to learn how oil shale is being mined and retorted and how shale oil is refined in countries where these operations have been conducted on a plant scale for many years. The authors spent 2 weeks at the plant of National Oil Proprietary, Ltd., at Glen Davis, and were afforded every opportunity to obtain detailed and specific information regarding all aspects of the company's operations.

### Units of Measure

For the convenience of readers, quantities are stated, unless otherwise indicated, in terms that are used in the United States, rather than in Imperial gallons, long tons, and Australian currency. An Imperial gallon is 1.20 U. S. gallons, and a long ton is 1.12 short tons. Therefore, an Imperial gallon per long ton is equivalent to  $\frac{1.20}{1.12} = 1.071$  U. S. gallons per short ton. Monetary values have been converted on the basis of the exchange rate in September 1947, which was \$3.25 an Australian pound.

### SUMMARY

This account describes the equipment and operations of National Oil Proprietary, Ltd., at Glen Davis, New South Wales, Australia, for mining and retorting oil shale and refining shale oil. The company is the successor of several organizations that

have attempted to commercialize the oil-shale deposits of New South Wales, first in the nearby Newnes area and later at Glen Davis.

At the time the plant was visited by the authors of this report (September 1947), operations were on a scale of 400 short tons of shale per calendar day. It was evident that the mine could produce a greater tonnage of shale if a larger force of mine employees could be maintained, and the retorts and refinery could handle double the quantity that was being processed. The refinery had been shut down about 200 days a year because of lack of oil, and usually only about half of the retorts were in operation, for lack of shale.

The mine is mechanized and is operated in a modern manner. The room and pillar method is being used. The main seam probably is the richest stratum of oil shale being mined in the world. It has a Fischer assay value of approximately 140 U. S. gallons per short ton.

The retorting plant consists of 104 Fell retorts of unique design, which have been evolved from Pumpherstons retorts originally erected at Newnes.

The refining equipment comprises a Dubbs 2-coil thermal cracking plant of 1,600 to 2,000 barrels a day capacity, a 350 barrels-a-day reforming unit, a stabilizer, and a Universal Oil Products high-pressure phosphoric acid polymerizing plant.

Stocks are treated by modifications of conventional methods with sulfuric acid, caustic soda solution, and doctor solution, and re-run to end-point requirements in a Foster Wheeler 2-stage atmospheric and vacuum still.

The principal marketed product is 70-71 octane (motor method) motor gasoline with 1.25 ml. tetraethyl lead per gallon. Small quantities of liquefied petroleum gases, cresols, and cracking-still coke are marketed, and fuel gas and oil are supplied for generating process steam and power.

The Glen Davis plant is a self-contained unit and has approximately 600 employees. On the whole, working conditions are good, insofar as housing and equipment are concerned. The village of Glen Davis is a company town that was built from the ground up for purposes of the industry. The site is isolated, which is a handicap in retaining employees who prefer to live in larger communities. However, morale is good among those employees to whom the isolated situation is not a major factor.

#### ACKNOWLEDGMENTS

This report has been prepared under the general supervision of W. C. Schroeder and R. A. Cattell, Synthetic Liquid Fuels Branch, Washington, D. C.

The authors are indebted to many officials of the Commonwealth and New South Wales governments and to members of the staff of National Oil Proprietary, Ltd. They are indebted, also, to members of the foreign service and the consular service of the United States in Sydney, Canberra, and Melbourne, Australia. The following are mentioned for their specific assistance to the authors. Many others, too numerous to mention here, provided information and analyses and otherwise contributed materially to this report.

E. J. Kenny, formerly chairman and managing director, National Oil Proprietary, Ltd., and now chairman of the company, was very cooperative, helpful, courteous, and cordial and spared no effort to enable us to obtain full information regarding the operations at

Glen Davis. L. J. Rogers, formerly assistant and deputy managing director of the company and now Commonwealth Fuel Advisor, was the source of a wealth of information from his long experience and wide knowledge of oil shale in Australia and throughout the world. He reviewed the manuscript and supplied many clarifying details. C. J. Norcross, formerly carbonization superintendent and now production superintendent, supplied many details of the operations at the plant, especially of the retorts, and contributed much background information regarding the history of oil-shale operations in New South Wales. The section on the shale industry of New South Wales is a slightly modified version of an account supplied by him, which in turn was derived largely from the monumental work of Carne.<sup>3/</sup> G. E. Mapstone, chief chemist at Glen Davis, was an excellent source of information on laboratory methods of analysis and plant control and furnished copies of his published and unpublished reports.

Others who were very helpful in supplying information and explaining the operation of the mine and plant were Sydney Christie, formerly chief engineer and now general manager, Les. Moore, formerly mine manager, and B. A. Raper, formerly refinery superintendent.

Special thanks are due to R. H. Crampton, purchasing agent of the company in Sydney, for obtaining hotel and travel reservations, making appointments, and many other acts of kindness and hospitality.

Senator the Honorable W. P. Ashley, Minister of Supply and Shipping, was much interested in our mission and extended many courtesies.

Knowlton V. Hicks, United States consul at Sydney, and members of the consulate staff gave valuable advice and assistance, such as clerical and secretarial services, and were especially helpful in arranging for the transportation of samples of oil shale and shale oil to the United States.

Acknowledgment is due H. P. Rue, chief, Fuels Technology Division, Region IV, and to the personnel of the Oil-Shale Research Branch at the Petroleum and Oil-Shale Experiment Station, Laramie, Wyo., for the analyses of the many samples of oil shale, shale oil, and products provided by National Oil Proprietary, Ltd.

The illustrations were redrawn from prints supplied by National Oil Proprietary, Ltd., under the supervision of L. F. Perry, supervising engineer of the Graphic Services Section of the Bureau of Mines, Pittsburgh, Pa.

The manuscript has been reviewed by E. D. Hardison, Simon Klosky, and Lester W. Schramm, of the Bureau of Mines, each of whom contributed valuable suggestions for improvement.

#### SUMMARY OF THE SHALE INDUSTRY OF NEW SOUTH WALES

In 1938, Kurth and Rogers<sup>4/</sup> reviewed the history and described plant-scale developments of oil shales in Australia. They stated:

- 
- <sup>3/</sup> Carne, J. E., The Kerosene-Shale Deposits of New South Wales: Memoirs, Geological Survey of New South Wales, Geology No. 3, Department of Mines and Agriculture, Sydney, Australia, 1903, XV plus 333 pages, 10 maps, 26 plates.
- <sup>4/</sup> Kurth, E. E., and Rogers, L. J., The Oil Shales of Australia. Oil Shale and Cannel Coal: Proceedings of a Conference held in Scotland, June 1938, pp. 193-209, The Institute of Petroleum, London, 1938.

Although oil shale has been discovered in Australia outside the borders of New South Wales and Tasmania, it is only within these two states that the known deposits, by reason of their extent and quality, are of any commercial interest. New South Wales and Tasmanian shales are of the same geological age, but entirely different in their physical and chemical characteristics. The former resembles closely the original Scottish torbanite, and occurs capriciously in small isolated deposits, frequently associated with bituminous coal, cannel, and carbonaceous shale. In Tasmania rich shale is found in only insignificant quantities. The main deposit consists of inferior shale, which yields, on distillation, an asphaltic oil containing much sulfur. The seam is more uniform, extensive, and accessible than those which have been discovered in New South Wales. It is for this reason, largely, that continued interest has been shown in Tasmanian shale, in spite of its disabilities.

### Discovery

Probably the earliest reference to the occurrence of oil shale in Australia was published in Paris in 1807 after the return of a French expedition from Australia in 1802. In "Voyage de Decouvertes aux Terres Australes" mention was made of "masses of 'schist bitumineux' which burned with a lively flame, giving off thick smoke and an odour of bitumen." This was found "at the foot of the mountains near Parramatta." It is probable that these "masses" were found where the Colo River leaves the mountains, in which case it is likely that they were originally part of the Newnes deposit (fig. 1).

In 1854, oil shale was exhibited at the Paris Exhibition among the products of New South Wales and stated to be from the River Lett, near Hartley.

In 1862 oil shale from Murrurundi was exhibited at the London International Exhibition.

The Sydney Morning Herald of July 14, 1865, records "a proposed undertaking to distil oil from kerosene shale at Stony Creek, near Maitland, and to refine the oil in an old Sugar Mill at Lavender Bay." However, some doubt exists whether this was kerosene shale or coal.

From this time, oil shales were discovered in many other localities, notably at Marangaroo, the Capertee Valley, Katoomba, the Wolgan Valley, Joadja, Torbane, Wollar, Barigan, Baerami, and Rylestone.

### Early Exploitation

#### America Creek

The Reverend W. B. Clarke, a geologist of the period, records that the "Pioneer Kerosene Works" at America Creek, Wollongong district, was the first to produce oil from colonial kerosene shale, the first shale being retorted in December 1865. This company used "D" retorts, with a capacity of 2 long tons a day. The oil was sold for 5 shillings, 6 pence an Imperial gallon.

#### Hartley

John Mackenzie, examiner of Coalfields, records oil recovery from Hartley shale in 1865.

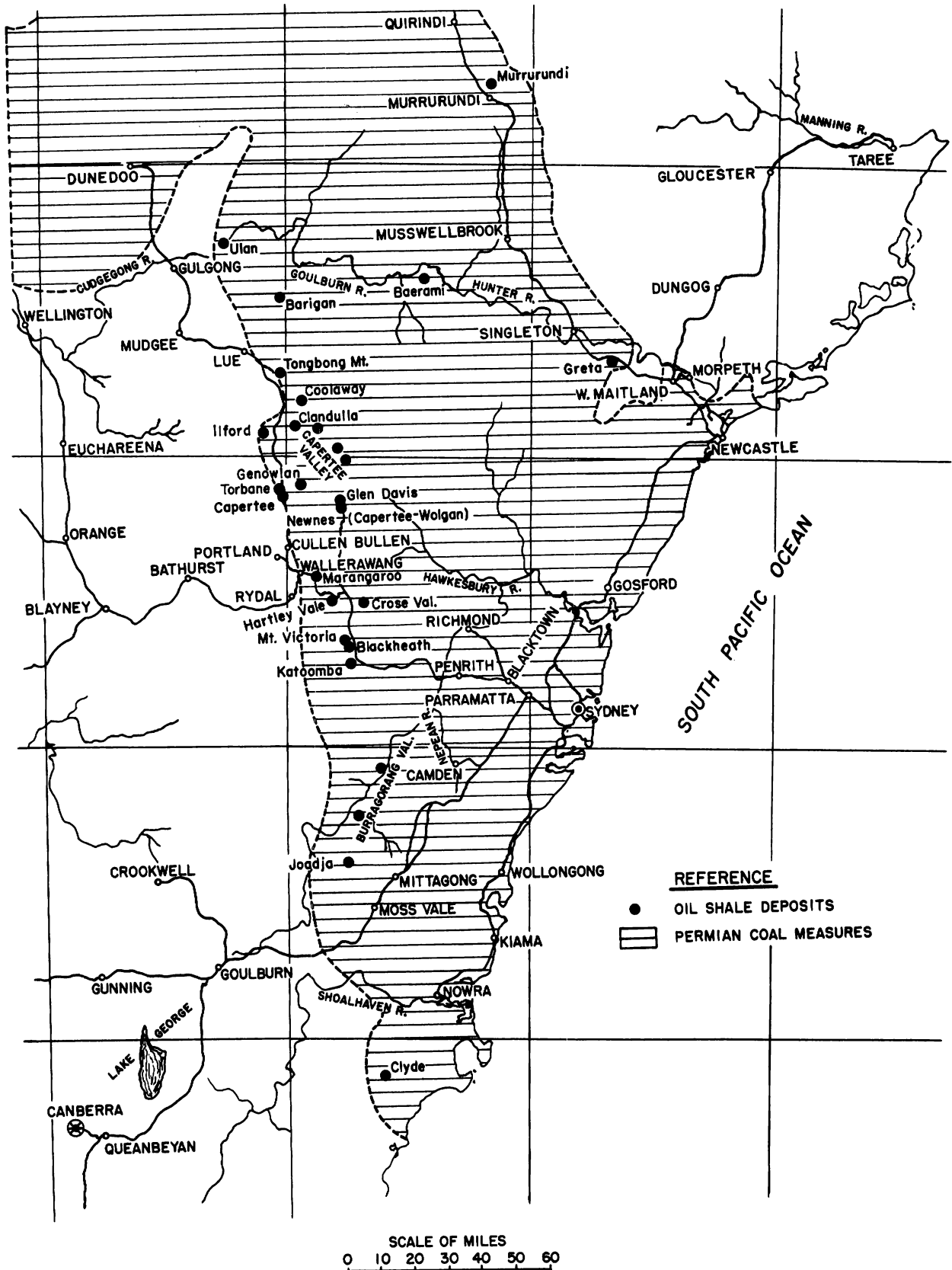


Figure 1. - Oil-shale deposits in New South Wales, Australia.



C. Humfrey, of the St. David's Oil Works, Saltney, England, writes of an examination of a shale sample from The Hartley Mine, saying, "It is the richest sample of oil mineral I have ever seen, yielding 150 gallons to the ton." He appends the following analysis:

58.5 percent condensible hydrocarbons  
12.6 percent ash  
9.5 percent fixed carbon  
19.4 percent gas and loss

W. Hall, one-time manager of the New South Wales Shale Oil Co., stated that the first retort tried at Hartley was a circular form fitted with trays, upon which the crushed shale was placed, as in a muffle furnace. A similar retort was erected at Kerosene Vale, near Lidsdale, by Mackenzie Bros., who used shale from the seam known as the Bathgate or Marangaroo.

The Hartley Company subsequently tried a retort on the principle of a lime kiln, and a similar retort was erected at Kerosene Bay, Port Jackson. Both of these retorts had been worked before 1868.

In 1868, the Western Kerosene Oil Co., also operating on the Hartley seam, erected a retorting plant of the "D" or gasworks type at Botany Road, Waterloo, having a capacity of approximately 500 pounds per retort, in benches of five retorts. These were found expensive, so units holding approximately 2,500 pounds were designed on the same principle and erected by John Glover, engineer to the company. Only the rich shale was retorted, the "seconds" or "bottoms" being rejected at the mine. Vertical retorts designed by W. J. Hall and J. Palmer<sup>5/</sup> were installed later to handle the "seconds." These were the first vertical retorts built in New South Wales.

#### Torbane

New South Wales Shale Oil Co. was formed in 1871 by combining the interests of the Western Kerosene Oil Co. and the Hartley Co., but the cost of shale extraction having increased, and the area of available shale at Hartley having diminished, the company commenced exploiting the deposit at Torbane, near Capertee, in 1896. Mr. W. Hall was sent to Scotland to select a retort suitable for processing this shale. He reported that the Scottish-retorts were unsuitable, and it was decided to erect a bench of Hall-Palmer retorts similar to those at Hartley<sup>6/</sup> but double the height and holding 15.7 tons, with a throughput of 3 tons daily, and charged with shale of 43 to 64 U. S. gallons per short ton assay value. The crude oil thus produced was forwarded to the Hartley refinery. Because of various difficulties, the works closed down, and the Commonwealth Co. later purchased the plant of the New South Wales Shale Oil Co., and erected a half bench of Bryson (or Pumpherston) retorts at Torbane.

#### Newnes

Because of the small extent of the shale deposits at Torbane, the Commonwealth Oil Corp. turned its attention to Newnes and in 1906 began the erection of a bench

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<sup>5/</sup> Work cited in footnote 3, plate VII.

<sup>6/</sup> Work cited in footnote 3, plate VIII.

of the Pumpherson retorts, which had been found fairly satisfactory on Torbane shale of poor or indifferent quality. The plant commenced operations in 1911 and closed after a few months. The reason given was that the rich shale so affected the retorts that operation was very difficult. John Fell assumed control as the representative of the debenture holders and subsequently formed his own company to acquire the business. He retained as his designing engineer William Petrie, previously of the Commonwealth Oil Corp., and 32 of the retorts were reconstructed in a manner designed to overcome the difficulties previously experienced. Some of the retorts were operated until 1922, when various costs were so high that the plant was closed again.

#### Joadja

Shale was discovered in the Joadja Valley near Mittagong about 1850 and was first worked in 1873, when the material was sold to the Australian Gas Light Co. at 3 pounds per long ton for gas enrichment.

Operation of "D" type retorts was begun about 1881. The works occupied 7 acres and supported a thousand people in 1888. The retorts were 66 in number and were stated to have a daily capacity of 5,600 pounds of shale each, but the cycle of charging is not known.

Four classes of products were obtained, namely, kerosine and candles for lighting, light "cleansing kerosine", wood-preserving oil, and lubricating products.

An experimental vertical retort was tried, but no general conversion to this type was made. The date of closing of the works is obscure, but it probably was closed some years before 1900.

#### Mornington

About 1900, a lead bath retort was set up at Mornington, but it did not operate beyond the experimental stage.

#### Murrurundi

In 1910, the British Australian Oil Co. erected a small bench of Scottish retorts at Murrurundi, and some oil was produced, but this plant was closed after a very small quantity of oil had been recovered.

#### Products

The first operations were largely for mining shale or torbanite to be used for manufactured-gas enrichment and to improve the luminosity of the gas flame for lighting. Shales were exported to the Netherlands, the United Kingdom, Italy, and the United States for this purpose until about 1911, when the introduction of the incandescent mantle made such use unnecessary. Kurth and Rogers<sup>7/</sup> estimate that 560,000 long tons were sent abroad from New South Wales to 1911, and approximately an additional 150,000 tons were used during the same period by Australian gas companies and railway departments. Although the best grade of shales was sold for gas manufacture, quite often the "seconds" were retorted at plants near the mines, and the oil was used for making paraffin wax and lighting kerosine, both of which commanded high prices. As the most important and profitable product of distillation was lamp oil, the torbanite came to be known locally as "kerosine shale."

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<sup>7/</sup> Work cited in footnote 44, pp. 196-197.



Kurth and Rogers estimated that the total amount of shale retorted for the recovery of oil in New South Wales up to 1922 was approximately 1,200,000 long tons. Refining operations to that time had been by distillation in small shell stills and coking in pot stills. The main products had been naphtha, lamp kerosine, gas oil, wood-preserving oils, greases, and paraffin wax for candle making. Because of the increasing market demand for motor fuels rather than lighting oils, this type of refining equipment became obsolete. In 1924, John Fell erected a 1,000-barrel-per-day cracking unit at Sydney to refine shale oil from the Newnes plant, which he had reconstructed some years previously. Because of financial difficulties, the refinery passed into the hands of the Shell Co., which used it for topping imported crude petroleum.

#### Recent Developments

From 1922 to 1940, little more than experimental work was done on oil-shale development in Australia.

#### Crown Ridge

In 1922 a small plant was erected at Crown Ridge, Capertee, by Vidler & Co., who installed a "Stockhausen"<sup>8/</sup> retort and worked the shale seam situated beneath the cap of the hill to the west of the Main Mudgee Road, a mile south of Capertee township. The deposit was small, and the venture was abandoned.

#### Torbane

In 1924, the Torquay & Anglesea Oil Co. was formed, and a plant based on a "Schultz"<sup>9/</sup> retort was erected at Torbane Siding. Shale was conveyed 3 miles by aerial tramway to the plant. Work on this project did not continue for long, and the installation was stripped and sold.

#### Wollar

Several years later, a Schultz retort was erected at Wollar by the Australian Imperial Shale Oil Co., but the project was abandoned without much progress having been made.

#### Mittagong

Early in 1930, a "Gotting"<sup>10/</sup> retort was erected at Mittagong, and small quantities of shale from Reedy Creek, Joadja, were carbonized. In 1941 a more ambitious plant of the same type was installed, but it proved unsuccessful and was replaced by a small setting of Dye-type<sup>11/</sup> retorts, shale being purchased from places as far afield as Barigan and Torbane.

#### Barigan

A small amount of rich shale has been won from this field to supply several small carbonizing plants in Sydney, Newcastle, and elsewhere.

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<sup>8/</sup> Stockhausen, Arnold Heinrich, and Vidler, William Henry, Shale Treatment; New Zealand Patent 47,316, Jan. 19, 1922.

<sup>9/</sup> Schultz, E. L., Australian Patents 14,182 (1923) and 16,966 (1928).

<sup>10/</sup> Gotting, H. E. B., and Gotting, L. K.: Australian Patents 109,341 (1939) and 113,575 (1940).

<sup>11/</sup> Dye, A. W.: Australian Patent 115,907 (1941).

## Newnes and Glen Davis

The exposures of shale in the Wolgan Valley (Newnes) and the Capertee Valley (Glen Davis) are part of the same deposit. In the Wolgan Valley, the seam was first opened by Campbell Mitchell in 1873, the site being later registered by Knoblanche and Wilkins as Portion 15, Parish of Gindantherie. The dates upon which the first shale leases were granted in the Glen Davis area are as follows:

M.P. 1 - Now worked by National Oil Proprietary, Ltd. -  
C. G. Cameron, 1881

M.P. 3 - R. Amos & Co. - May 27, 1897

M.P. 4 - R. Amos & Co. - May 27, 1897

M.P. 5 - R. Amos & Co. - May 27, 1897

M.L. 4, 5, 6, 7, 8, - John Lang - May, June, October 1895

Because the field was virtually exhausted in the vicinity of Newnes in operations up to 1922, all subsequent interest has centered in the Glen Davis area. In 1932, a grant was made by the Government for the relief of miners, and a portion of this money was spent in reoperating several of the retorts at Newnes with a view to proving the commercial value of the undertaking. When the allotted amount had been spent, the plant was closed. Upon the formation of National Oil Proprietary, Ltd., in 1937 much of the Newnes plant was dismantled and transported to Glen Davis. Figures 2 and 3 are photographs, taken in 1947 by the authors, of the remains of the Newnes plant, showing its former sturdy and substantial construction.

## Baerami

In proved tonnage the Baerami field ranks before but in potential oil yield next to Glen Davis. Half the seam by thickness (and more than half by weight) is composed of cannel coal, and the average assay value is approximately 64 gallons a ton. Attempts have been made since 1939 to develop the field on a large scale with Renco retorts (see p. 10), but the necessary capital has not been obtained. During World War II the leases were worked by Shale & Oil Products, Ltd., which produced a small quantity of oil in Gotting retorts at Sand Hollow.

Each of the 20 to 30 known deposits of oil shale in New South Wales is believed to be small. Only two - those at Glen Davis and Baerami - have been proved to be large enough (perhaps 9,000,000 tons at Glen Davis and 14,500,000 tons at Baerami) to justify operations even on a small scale, such as would result in the production of 700 barrels of gasoline a day.

## Newnes Investigation Committee

In February 1933, a committee known as the Newnes Investigation Committee, composed of representatives of the commonwealth and New South Wales state governments,<sup>12/</sup> "inquired into the possibility of developing the shale-oil industry in the Newnes-Capertee area on a sound basis." Investigations of the committee included a study of

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<sup>12/</sup> Report on the Newnes-Capertee Shale-Oil Project by the Newnes Investigation Committee to Senator A. J. McLachlan, Minister in Control of Development for the Commonwealth of Australia, and to R. S. Vincent, Minister for Mines of the State of New South Wales, 1934.

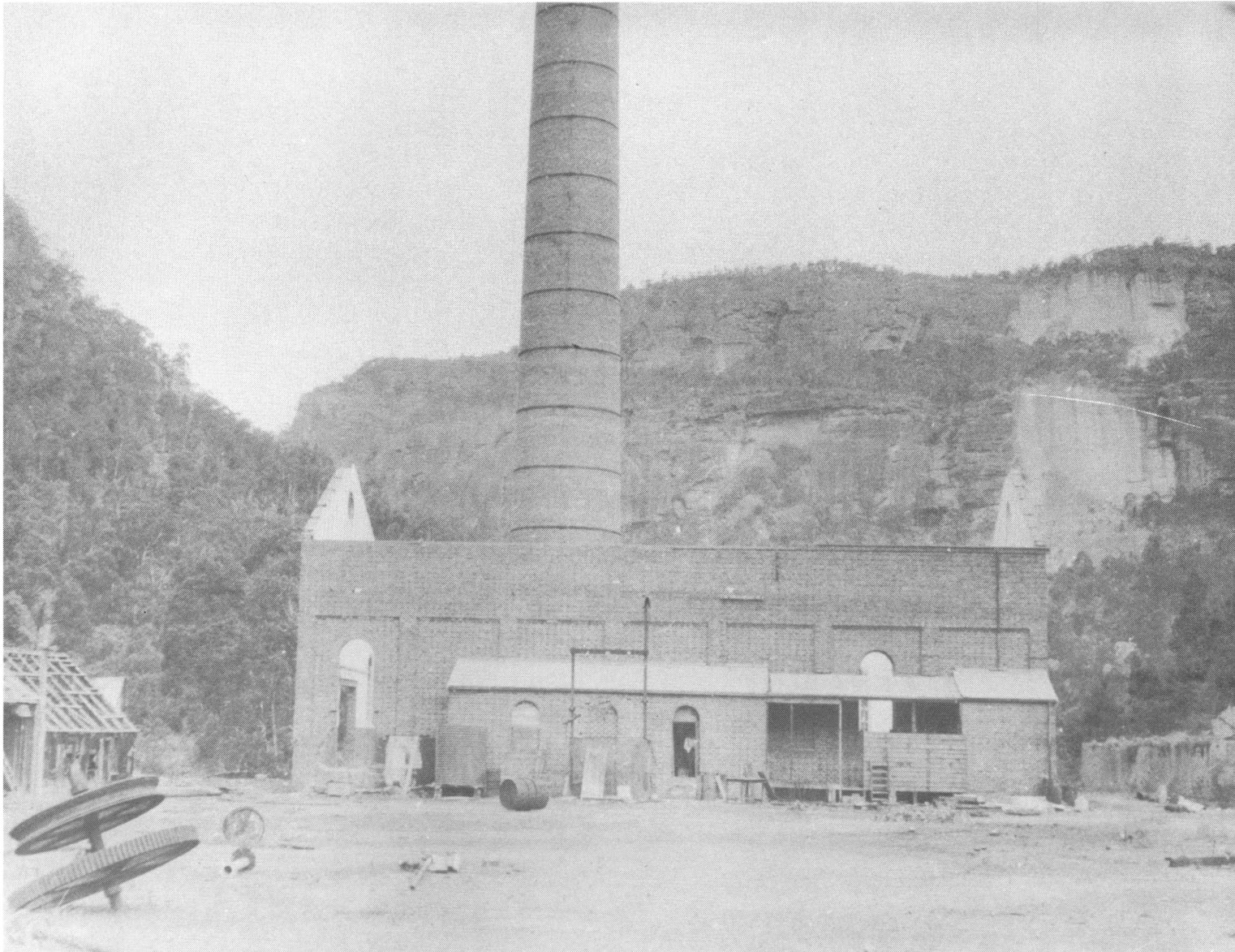


Figure 3. - Ruins of power plant, Newnes, N. S. W., Australia (September 1947).



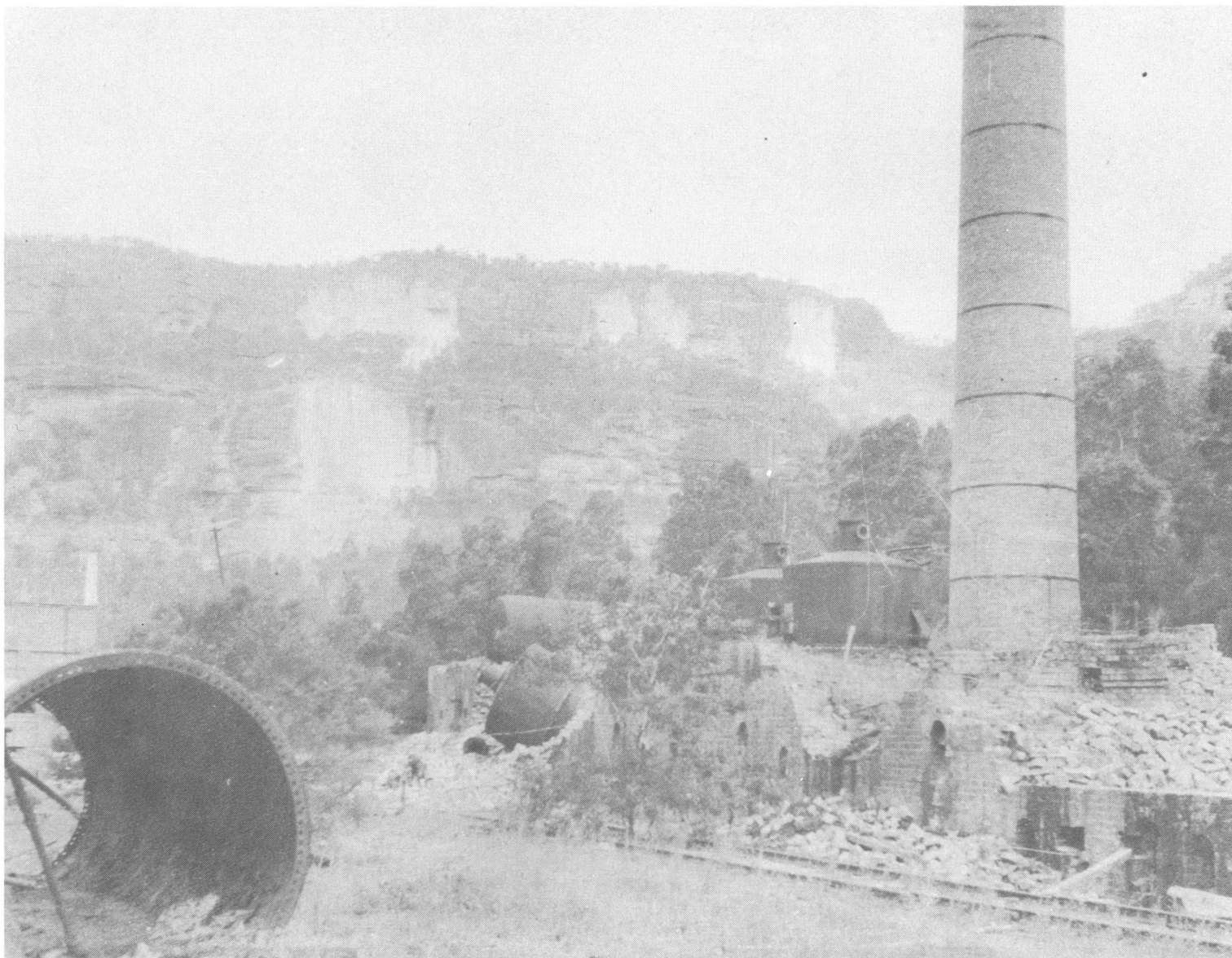


Figure 2. - Ruins of pot and shell stills, Newnes, N. S. W., Australia (September 1947).



methods for refining crude oils from the Newnes-Capertee shales. L. J. Rogers, Commonwealth Fuel Research Officer, was sent to Great Britain and the United States to inquire into the retorting of shale and refining of crude oil. He interviewed manufacturers of refinery equipment in England, inspected retorting and refining plants of the Scottish oil-shale industry, and obtained information from Imperial Chemical Industries, Ltd., on the production of motor gasoline by hydrogenation. In the United States he witnessed pilot-plant cracking tests on Newnes shale oil by the Universal Oil Products Co., the M. W. Kellogg Co., and the Winkler Koch Co. and discussed various problems arising in the cracking of shale oil and the chemical treatment of cracked products.

From the results of these and other investigations and tests, the committee was able to evaluate different methods of refining Newnes-Capertee crude shale oils:

1. The yields of motor fuel and residuum by cracking the crude oil to fuel oil, pitch, or coke; the advantages of reforming the straight-run naphtha; the capital and operating costs involved in cracking the crude oil, reforming the naphtha, treating the crude gasoline, and blending tetraethyl-lead with the finished product.
2. The yield of motor fuel from the crude oil by catalytic hydrogenation; the cost of installing and operating the necessary plant.
3. The cost of producing various refined-oil fractions by straight distillation.
4. The yield and cost of various products obtained by a modified system of cracking, intended primarily for the preparation of a fuel oil by breaking down the waxes.
5. Storage facilities required for crude oil and finished products.
6. The disposal of residues and recovery of sulfuric acid.

After evaluating the various refining processes and considering other factors, such as market demand, transportation, investments, etc., the committee recommended that the crude oil be refined by cracking to motor fuel and fuel oil.

A company, known as National Oil Proprietary, Ltd., was formed by private interests with financial assistance provided by the governments of the Commonwealth and New South Wales. Much of the retorting and other plant equipment was moved to Glen Davis from the Newnes plant, and new refinery equipment was purchased from the United States. The Glen Davis plant began operations early in 1940. Refining and other operations of the Glen Davis plant have been described at various periods since that time; W. T. McFadyen in 1941,<sup>13/</sup> J. M. Antill in 1944,<sup>14/</sup> and R. F. Cane in 1947.<sup>15/</sup>

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<sup>13/</sup> McFadyen, W. T., Refining of Shale Petrol: The Modern Engineer, vol. 15, No. 3, Mar. 1941, pp. 96 (Melbourne).

<sup>14/</sup> Antill, J. M., The Shale-Oil Industry in New South Wales: Chem. Eng. & Min. Review, vol. XXXVII Oct. 10, 1944, pp. 10-18 (Melbourne).

<sup>15/</sup> Cane, R. F., Production of Motor Fuel from Oil Shale: The Modern Engineer, Nov. 20, 1947, pp. 221-25 (Melbourne).

### American Shale Mission

The Oil Shale Mission of the United States Board of Economic Warfare,<sup>16/</sup> which visited Australia in 1942, recommended to the Commonwealth Government a much more ambitious program of expansion for Glen Davis and Baerami. Each of the projects, as recommended by the Mission, would have produced 30,000,000 to 36,000,000 gallons of oil products a year. The proposed operations were contingent to a large extent upon the use of Renco retorts.<sup>17/</sup> National Oil Proprietary has constructed a pilot retort of that design with a view to obtaining further information about the process before a final decision is made on the Mission's recommendations. Figure 4 is a photograph of the Renco pilot plant at Glen Davis taken in September 1947, before construction was completed. Figure 7 shows the location within the plant, near the Capertee River.

Tests were made at Newcastle, New South Wales, in 1939, by an Australian engineering organization, on a Renco retort of the same size as the one at Glen Davis. In fact, the retort itself (the vertical cylindrical structure in the center of the picture, figure 4, the special valves for charging the raw shale and discharging the spent shale, and the heat exchanger-condenser of the Glen Davis unit were originally part of the retort in which the tests were made at Newcastle in 1939. When the plant was erected at Glen Davis, the retort was lengthened by insertion of a section 6 feet, 6 inches long, the heat exchanger-condenser was altered slightly, and a number of other alterations were made.

The Renco retort is a continuous apparatus in which the shale moves vertically by gravity through the retort in the presence of hot circulating product gases under a pressure of about 200 pounds per square inch. The equipment comprises (1) the retort itself and means for charging and discharging it under pressure, (2) a system of pipes, compressors, and a furnace for circulating hot product gases under pressure, and (3) heat exchangers and condensers for liquefying product vapors. In figure 4 the central portion of the plant is the lengthened retort, with the tube furnace for heating the circulating gases in the background. The white building in the background contains the control instruments, the gas compressors, and transfer pumps. The large rectangular structure at the right of the retort is the raw-shale storage bin or charging hopper, and the shale elevator is shown at the right end of the charging hopper. Shale for charging the retort is unloaded from trucks into the concrete pit at the bottom of the elevator. The smaller elevated box at the left is the spent-shale hopper.

The retort was virtually ready to operate at the close of 1948, but because of shortage of personnel and funds, test runs have not been made.

### N-T-U Retorts

An operation of particular interest was conducted during the Second World War by Lithgow Oil Proprietary, Ltd., at Marangaroo, near the town of Lithgow, New South Wales, to augment the supply of liquid fuel for commercial uses. This company designed and operated three N-T-U retorts, mainly on the basis of information in Bureau of Mines Bulletin 315.<sup>18/</sup> Figure 5 is a view of the retorting plant at Marangaroo

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<sup>16/</sup> Oil and Gas Journal, Expansion of Shale-Oil Industry in Australia Depends on Lend-Lease Aid: Vol. 42, No. 34, Dec. 30, 1943, pp. 142, 145-6.

<sup>17/</sup> Jacomini Virgil Victor, Improved Method of, and Apparatus for, the Distillation of Oil from Shale or Other Carbonaceous Matter: Australian Patent 118,783, August 24, 1944.

<sup>18/</sup> Gavin, M. J., and Desmond, J. S., Construction and Operation of the Bureau of Mines Experimental Oil-Shale Plant 1925-1927: Bureau of Mines Bull. 315, 1930, 154 pp.



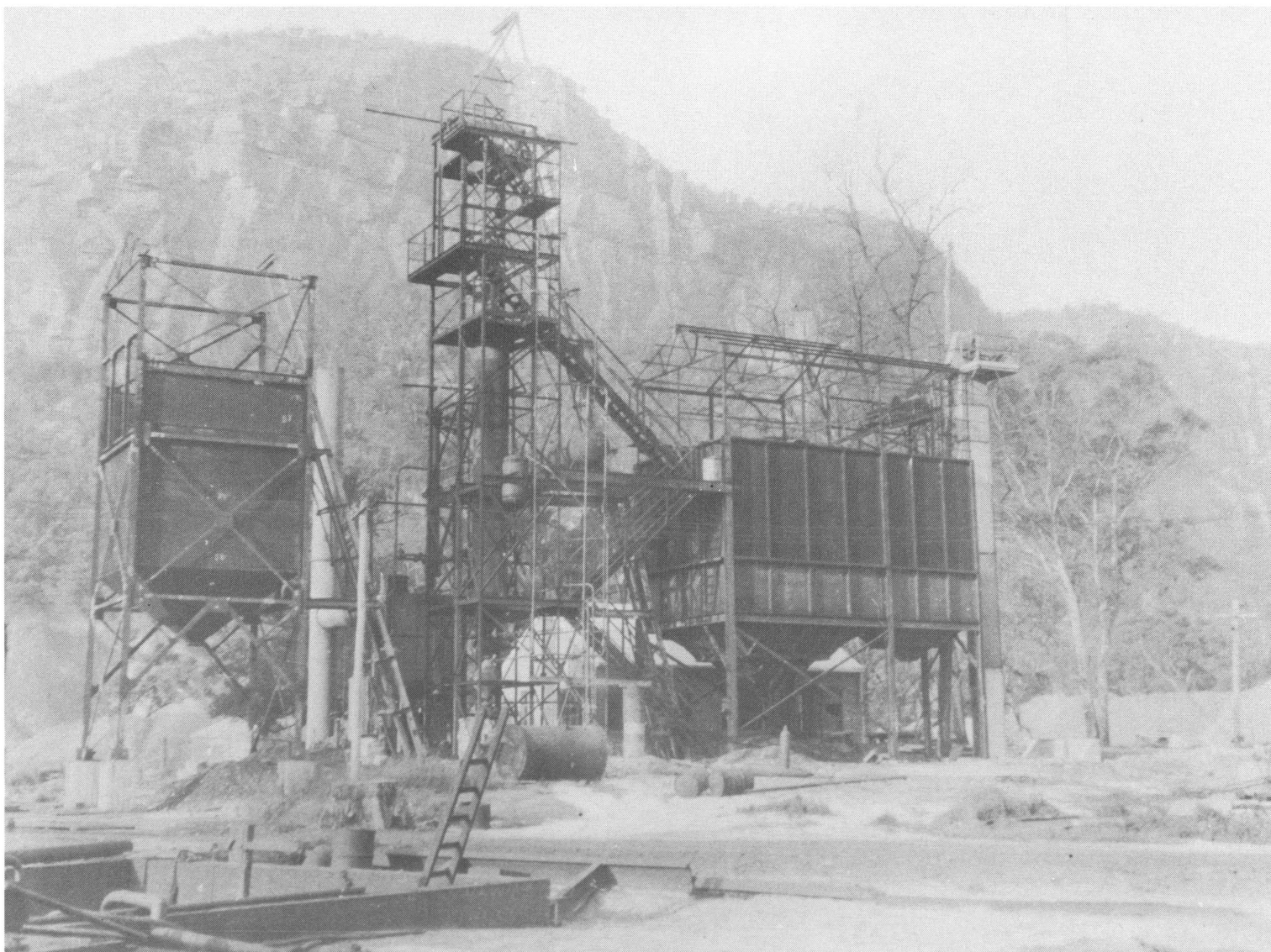


Figure 4. - Renco retort at Glen Davis, N. S. W., Australia (September 1947).



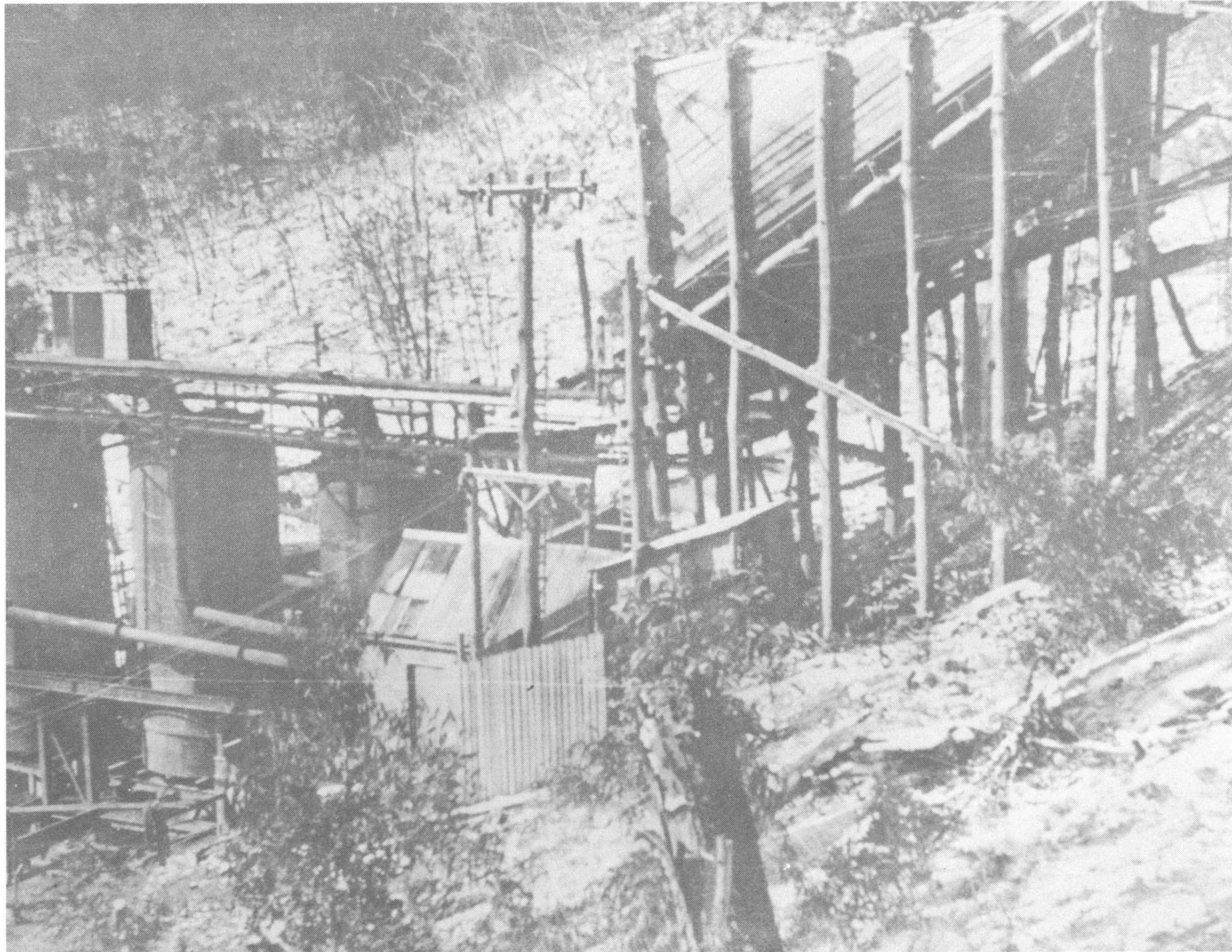


Figure 5. - N-T-U retorting plant at Marangaroo, N. S. W., Australia.



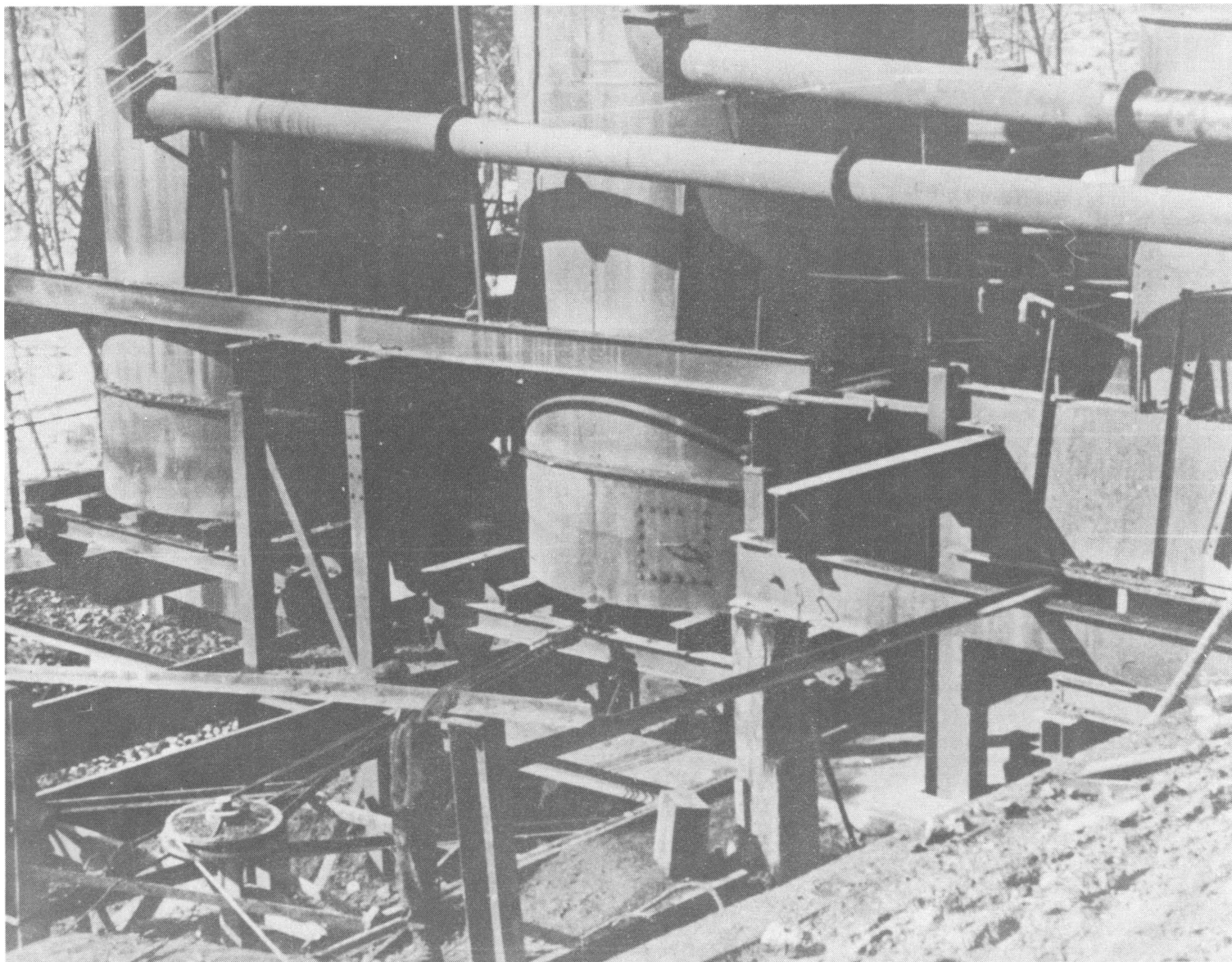


Figure 6. - N-T-U retorts at Marangaroo, N. S. W., Australia.



showing the three vertical N-T-U retorts below the crushed shale-storage bin and the crusher house. The portal of the mine was at the right of the area shown in the picture. Figure 6 is a close view of the retorts showing the winch ropes in position to pull the grate from the middle retort at the end of a run. Despite the primitive appearance of this plant, built under wartime restrictions and scarcity of materials for temporary operation, more than 2,000,000 gallons of crude shale oil were produced during the calendar years 1943 to 1945, inclusive. The oil was refined at the Glen Davis plant.

#### GLEN DAVIS OIL-SHALE PLANT

##### Organization of National Oil Proprietary, Ltd.

The operating company, National Oil Proprietary, Ltd., was organized in 1937. The company has a board of three directors, who are nominated by the governments of the Commonwealth of Australia and the State of New South Wales and approved by the ordinary (common) shareholders. Operation of the plant is partly dependent on financial assistance given in the form of remission of excise duty, amounting to the difference between the sum of a duty of 10 pence an Imperial gallon and approximately 0.9 penny primage, a total of 10.9 pence, which is levied against imported gasoline, and a duty of 3 pence an Imperial gallon, levied on gasoline made from indigenous material (oil shale, or benzol from coal). Therefore, the preference in favor of gasoline made at Glen Davis is 7.9 pence (approximately 10.7 cents) an Imperial gallon, equivalent to 8.9 cents a U. S. gallon.

The principal supervisory officials at Glen Davis are:

- Managing director
- Production superintendent
- Mine manager
- Chief engineer
- Refinery superintendent
- Chief chemist
- Secretary-accountant and office manager
- Property officer
- Industrial officer (personnel relations)
- Welfare officer (including safety)

This staff and their assistants appeared to be highly competent, interested in their work, and their esprit de corps was very good. The morale of the plant employees seemed to be high, insofar as personal relations were concerned, but would have been better if uncertainties regarding future supplies of shale and continuous plant operation could have been eliminated.

##### Village of Glen Davis

The village of Glen Davis was established at the time the oil-shale plant was built in 1938 to 1940. The village and plant lie in the narrow Capertee Valley surrounded by escarpments that rise sharply above talus slopes. The site is isolated, and there were few facilities for amusement or recreation. Therefore, the company originally provided a golf course, tennis courts, a bowling green, and children's playgrounds, which are now maintained by the citizens as a group. When the village was built, flowers and flowering shrubs were planted, and the area now has a very pleasing appearance.

At the time of the authors' visit (September 1947), the settlement had a population of 1,600 and comprised approximately the following living quarters:

1. Eleven substantial brick staff residences and a brick hostel that can accommodate about 30 junior staff employees.

2. One hundred permanent and 50 war-type individually owned residences, financed and constructed under cooperative building schemes.

3. A group of barracks with rooming and boarding accommodations for 300 single men, two in a room.

4. A commercial hotel of about 25 rooms.

5. A "Bag Town" of about 125 houses, mostly substandard.

A school, housed in three buildings, had 7 teachers and about 260 pupils. Classes ranged from kindergarten through junior high school.

The town had one physician and one druggist, three churches, and a community center.

Commercial establishments were few and included two general stores, a garage, a drugstore, a saloon, a barber shop, and a hotel. A moving-picture theatre in "Bag Town" must remain the only theatre in the area until a 3-year National embargo on construction of theatres expires.

The bank, post office, telegraph office, and police station are either Commonwealth or State institutions.

#### General Layout of the Plant

Figure 7 is a scale drawing of the layout of the Glen Davis works showing the relative position and size of the various above-ground units. The entrance to the mine is at the lower right of the drawing and can be oriented in relation to the other portions of the plant by reference to figure 8, which shows the entrance to the mine at the 1,110-foot contour line, the shale-storage bins, and the retorts south of the Works Road.

#### Oil-Shale Mine

As may be seen in figure 8, the mine is developed by the room and pillar method. The main portal of the mine, M.P. 1 tunnel, is at an elevation of about 1,000 feet above sea level.<sup>19/</sup> The shale beds dip toward the mine portal at approximately a 6-percent grade, thus aiding haulage from the mine. The seam outcrops in the talus slope previously mentioned and the four adits used for ventilation, access, and haulage are driven into the shale itself.

It is a modern, mechanized mine that uses electric power. Electric trolley locomotives operate on 250-volt alternating current. A motor-generator set converts 2,000-volt alternating current to direct current for charging shuttle-car batteries, and the high-tension current is stepped down to 440 volts for other purposes.

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<sup>19/</sup> The datum plane is approximately 100 feet below mean sea level.



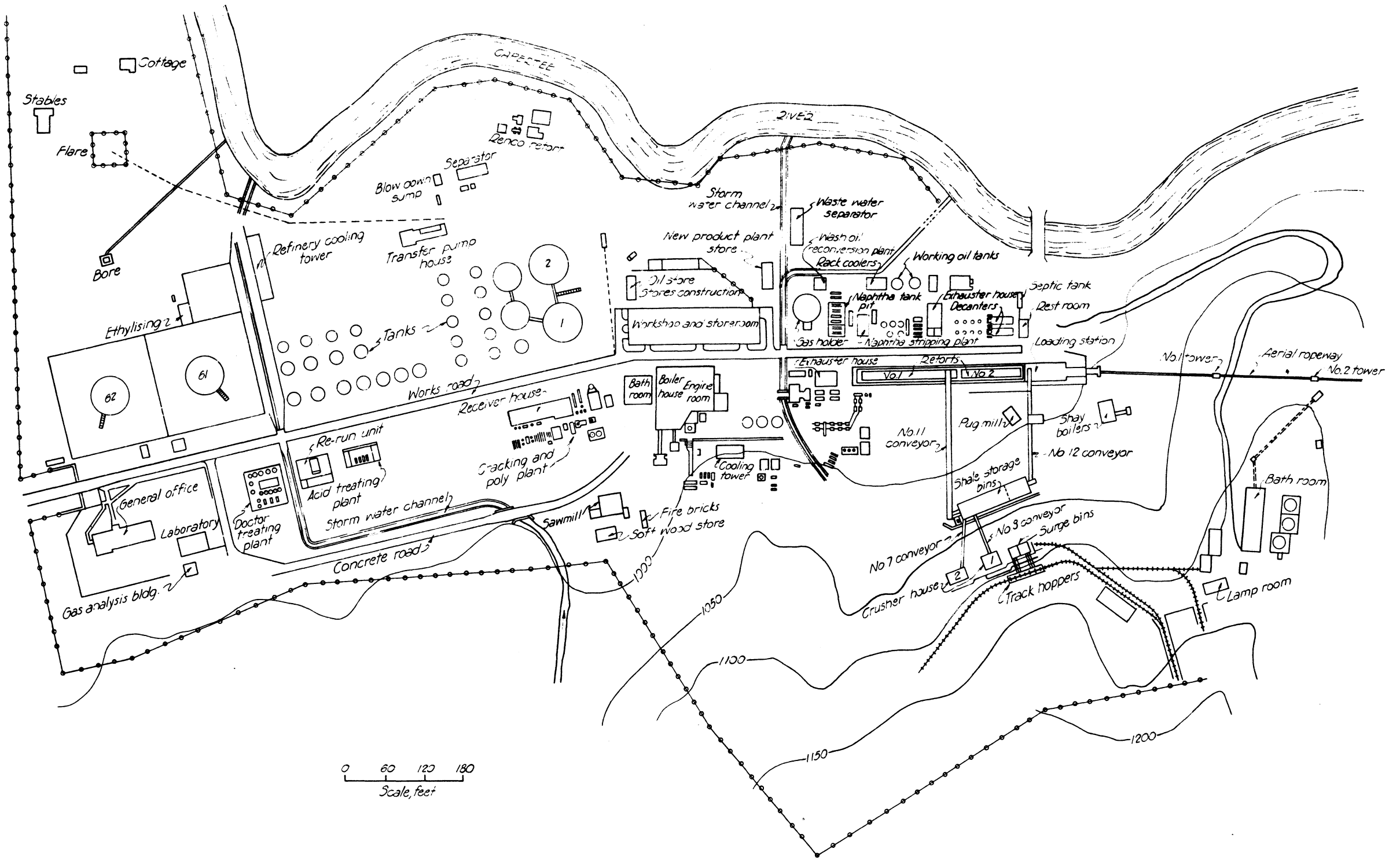


Figure 7. - General lay-out of Glen Davis works, National Oil Proprietary, Ltd., New South Wales, Australia.



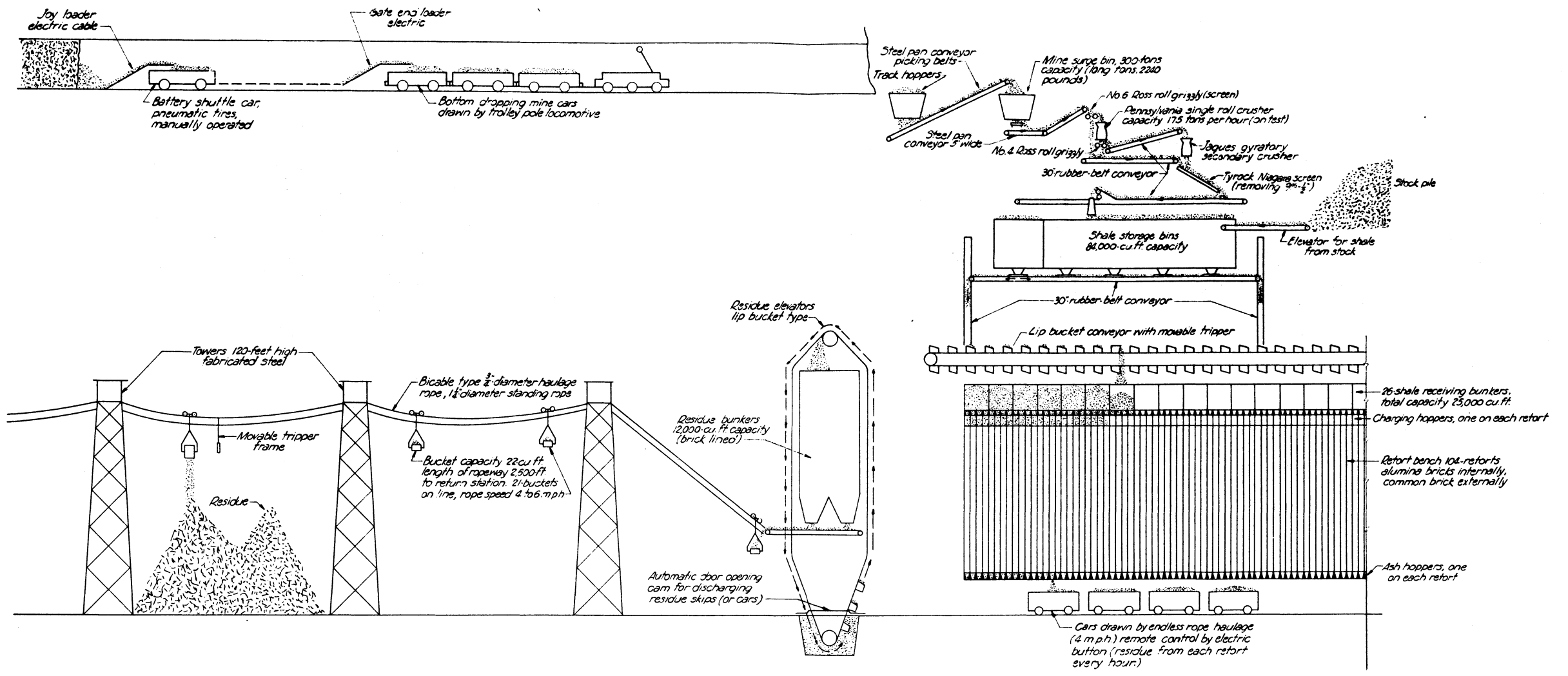


Figure 10. - Schematic drawing of the movement of shale from the mine face to the spent shale dump at Glen Davis.  
 (After: C. J. Norcross)



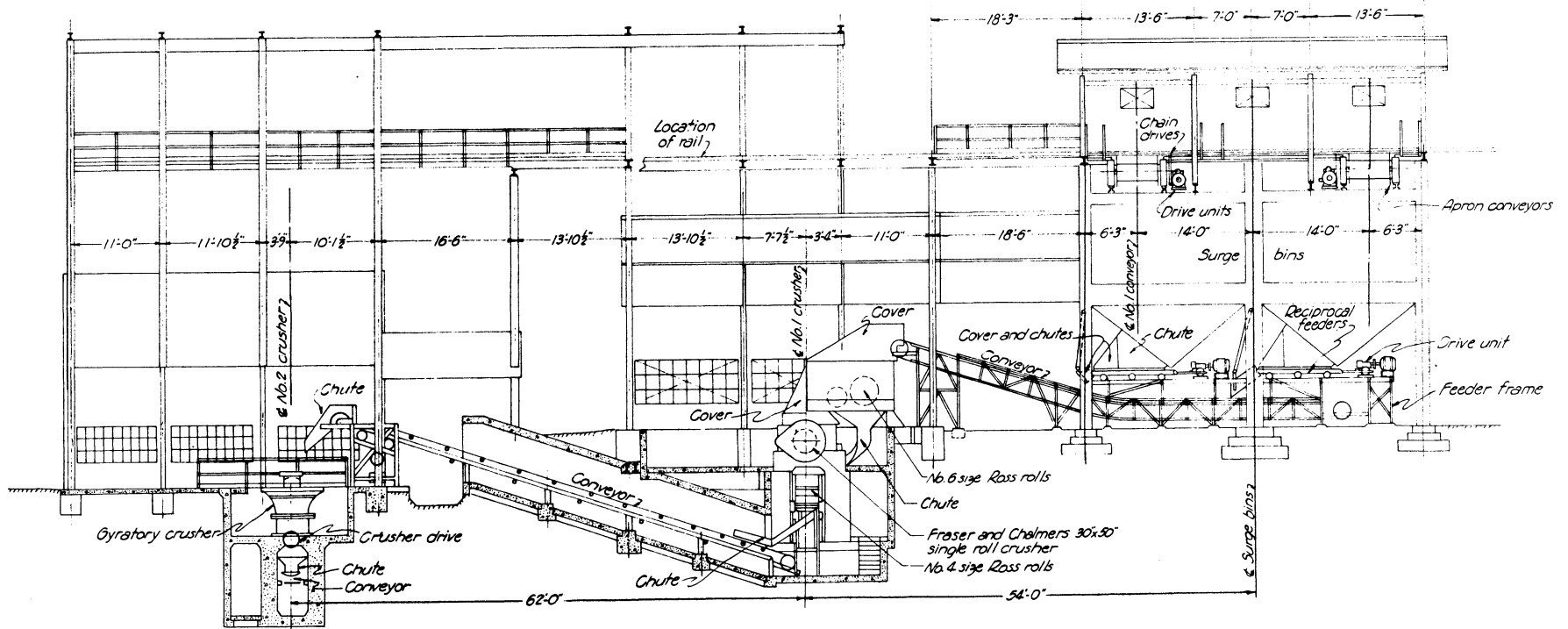
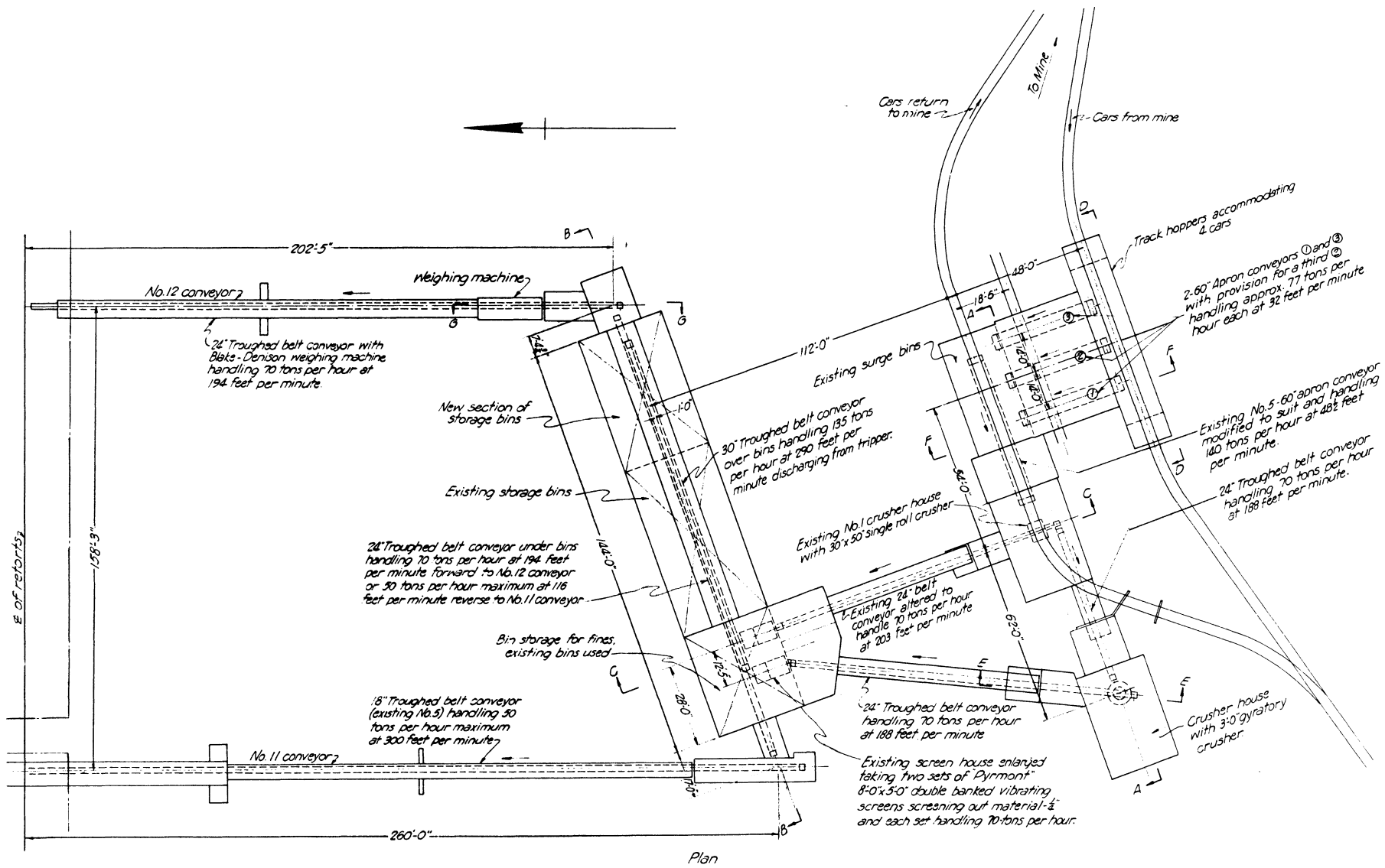


Figure 11. - Layout of shale-handling plant, Glen Davis, New South Wales, Australia.



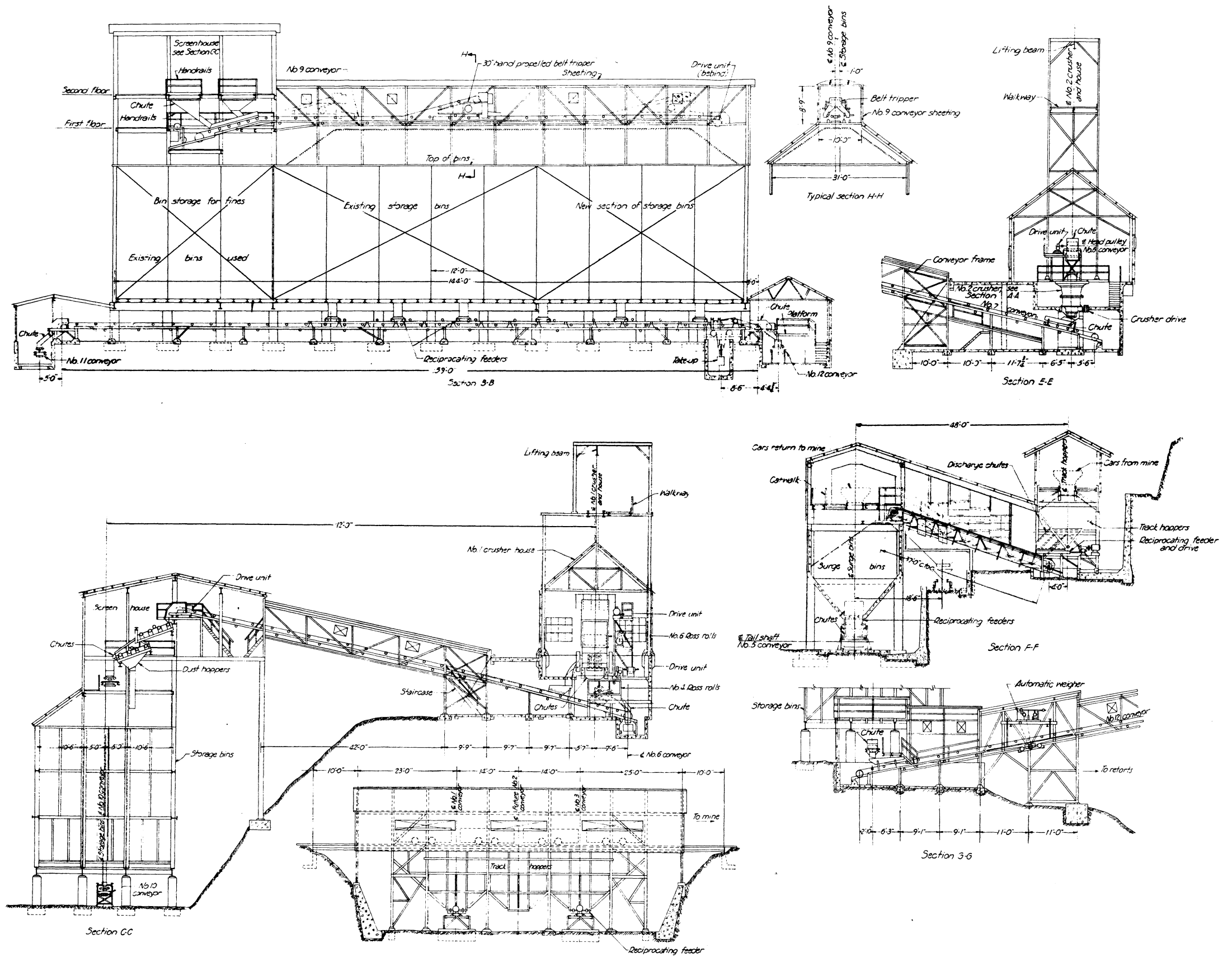


Figure 12 - Lay-out of shale-handling plant.





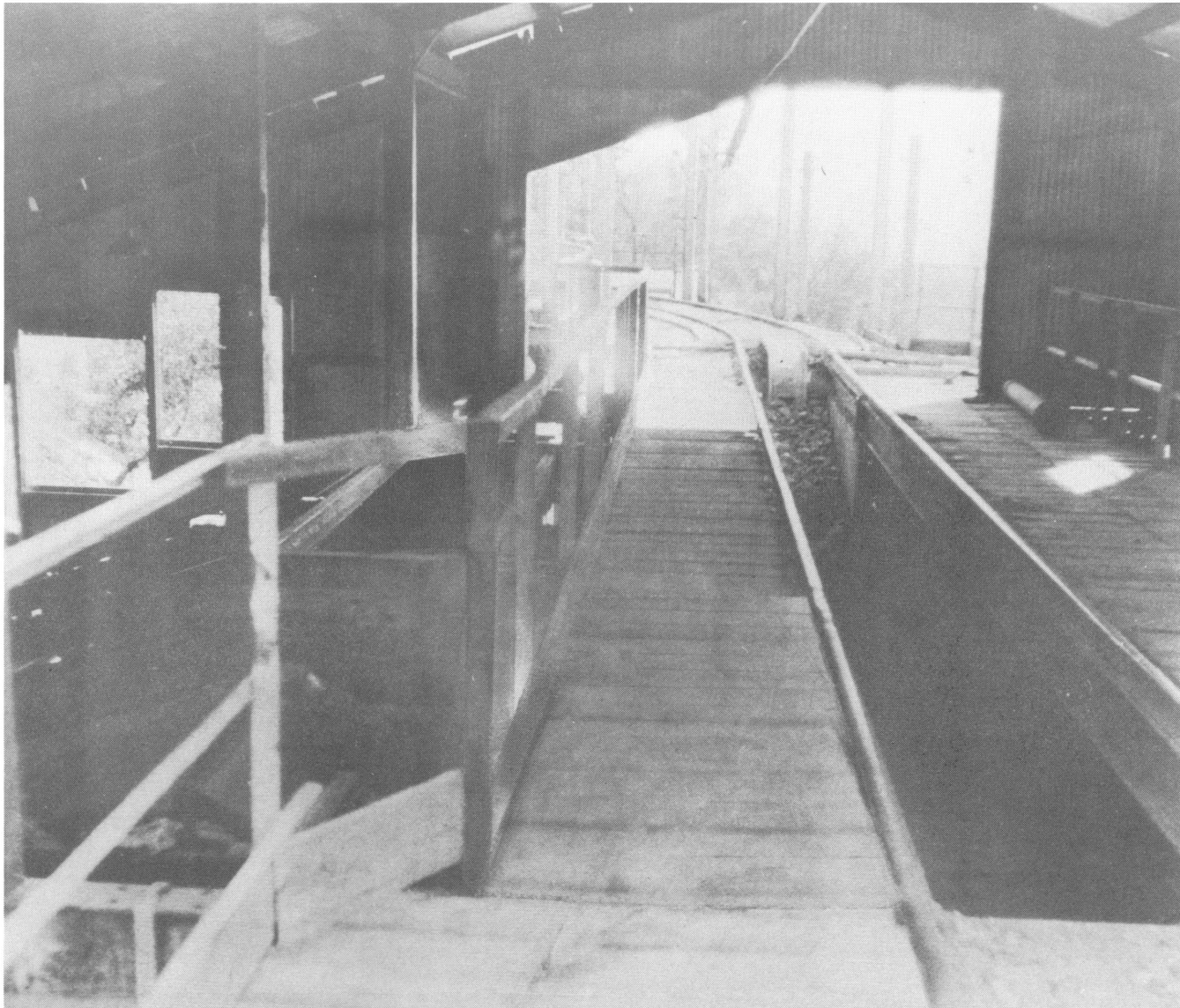


Figure 13. - Picking belts and bins, Glen Davis, N. S. W., Australia. Left; picking belts discharging into surge bins below gap between track rails. Right; direct line for emergency discharge of mine cars into surge bins.



This low productivity is said to be due in part to the room-and-pillar method of mining, which leaves 60 percent of the shale as pillars. The managing director expressed the opinion that the "long-wall" method of mining probably should have been adopted. Mine productivity should be higher during the time the pillars are being removed, particularly if mechanical removal is practiced.

The cost of net shale delivered to the crushers in 1947 was approximately \$4.15 a ton on the basis of \$3.25 an Australian pound. More than half of this amount was due to "face cost", that is, costs at the working face of the mine. The run-of-mine shale had an average Gray-King assay value of 64 gallons a ton.

The management hopes to be able to increase the output of the mine and improve its quality, so that the present retorts can be operated more nearly at capacity. The refinery was on stream only 70 days in the first 6 months of 1947 and could handle all the oil that the present two benches of retorts could produce if they were operated at maximum efficiency. The principal requirement for increasing mine output seems to be more miners and reduction of absenteeism, which averages 20 percent or more. The management also has hopes of developing a practical method of mining only the main (rich) seam, thus increasing the assay value of the charge to the retorts. They have little hope of finding a new and thicker rich seam.

#### Carbonizing Department

The following description of the equipment and methods of operation in the crushing and retorting plant has been adapted from an account written especially for this report by C. J. Norcross, production superintendent at Glen Davis. Mr. Norcross drew the sketch from which figure 10 was drawn, showing schematically the flow of shale from the mine face (upper left), through the crushers and screens to the retorts (right), and the travel of the spent shale from the retorts to the residue dump (lower left).

The Carbonizing Department encompasses the operation of crude-oil production from the receipt of oil shale from the mine to the delivery of crude oil and naphtha to the Refining Department.

#### Crushing and Screening Section

Shale is received (see figs. 11 and 12) into concrete surge bins of approximately 300 tons capacity, being then a mixture of all sizes from 1 foot by 1 foot blocks downward to particles that will pass through a 1/8-inch screen and in September 1947 contained enough kerogen to produce a little more than 64 gallons of crude oil and naphtha per ton in an assay retort. Its potential yield is influenced directly by the amount of carbonaceous shale and clay of low assay value (15.5 gallons a ton), which is mixed with the rich primary seam (135 gallons a ton) during mining operations.

The shale is fed from the bottom of the surge bins (fig. 13) by two reciprocating plate feeders powered by 7-1/2-horsepower electric motors, onto a steel apron conveyor 5 feet wide, powered by a 10-horsepower electric motor, which delivers to a No. 6 Ross rolls grizzly screen before entering the crusher. This screen is of the plain grooved-roll type powered by a 15-horsepower motor, succeeded by an eccentric clearing roll, which passes all shale larger than 5 inches into the crusher. The material smaller than 5 inches passes through the grooves of the roll and joins the crushed material as the latter leaves the crusher.

The crusher is a "Pennsylvania" single-roll type, in which a toothed roll 3 feet in diameter by 4 feet, 6 inches long crushes the shale against a back plate until the pieces are small enough to pass between the roll and the plate. The power is applied through a 125-brake-horsepower motor, and the unit has crushed at the rate of 195 tons of shale an hour under test.

Upon leaving the crusher, the shale falls upon a second No. 4 Ross rolls grizzly, which passes shale larger than 5 inches onto a rubber belt conveyor leading to the secondary crusher. The shale smaller than 5 inches passes through the grooves and joins the minus 5-inch shale from the first set of rolls upon a rubber belt conveyor delivering to the "Tyrock Niagara" screen, where the material smaller than 1/2 inch is removed, which amounts to about 11 percent of the mine run. The screen is of the vibrating type, with three decks of woven wire-screen cloths and delivers shale of 1/2 to 5 inch size to a belt conveyor, which, in turn, delivers to storage bins of 2,240 tons capacity.

The secondary crusher, which receives the oversize shale from the second set of Ross rolls grizzlies, is of the Traylor gyratory type driven by a 70 brake-horsepower electric motor. The crusher bearing oil is water-cooled. After being crushed, the shale passes over a rubber belt conveyor and joins the flow of "retortable" shale from the Tyrock Niagara screen, passing thence into the storage bins. A "Pyrmont"-type vibrating screen originally was fitted in the line of the flow of the recrushed oversize shale but was removed after trouble had been experienced repeatedly with broken springs; the fine material produced by the recrushing is now being left with the retortable shale.

Because of their height and the effects of back pressure, the Fell retorts are not suitable for a shale charge that contains a substantial proportion of fines, and this is the primary reason for the screening operation. However, removal of fines and dust is not as wasteful as might appear at first glance for the following reasons:

1. Poor material crushes more finely than the rich seam. Fines and dust assay about 35 gallons a ton, compared to 64 gallons a ton for run-of-mine shale.
2. Fines are being stock-piled with the intention of retorting them later. It is partly for this reason that the Company is interested in the Renco retort.
3. The poor material disintegrates and becomes slimy on exposure to weather, whereas the rich shale does not. Therefore, screening when the fines are reclaimed from the stock pile should yield shale of better quality.

If the mine output exceeds the retort requirements, the excess shale is removed from the storage bins by motor trucks and dumped on a stock pile of the bins.

If the shale supply from the mine is insufficient for retort requirements, a dragline scraper driven by a Sullivan winch draws shale from the stock pile and feeds into a bucket elevator, which delivers to the Tyrock Niagara screen previously mentioned. Material smaller than 1/2 inch is removed by the screen, and the remainder passes over the conveying system to the storage bins.

The section described is known as the Crushing and Screening Section and its present form was arrived at in the following manner:

It was known that Newnes oil shale presented certain crushing difficulties, and in 1938 a 30- by 50-inch Pennsylvania super-armor-frame single-roll crusher was

purchased. The crusher was to have a capacity of not less than 100 tons of oil shale an hour, taking pieces up to 36 by 24 by 18 inches and reducing them to a product 85 percent of which would pass a 4-inch ring. During the trial run it was found that comparatively small pieces of shale fed to the crusher caused the toggle bars to break. When these were strengthened, the shear pin that had been fitted to the wheel at the request of National Oil Proprietary gave similar trouble. The pin was strengthened, which allowed the crusher to function, although many pins continued to break. In 1945 the roll shaft was increased in diameter, and a still stronger shear pin was fitted. This has overcome the trouble, and broken pins are rare.

The original plant was not large enough to handle the volume of shale necessary to supply 104 retorts, so in 1945 a number of alterations were made in an endeavor to speed the throughput.

The form and extent of these alterations were governed by the fact that the old plant had to continue working during alterations. A secondary crusher and two Ross roll grizzlies were installed, together with a "Pyrmont" screen following the secondary crusher. New belts conveyors were installed, and the capacity of the existing belt was increased. Reciprocating plate feeders were fitted to the outlet of the surge bins, and the flight conveyor was raised to accommodate the Ross roll-grizzly screen.

#### Crushing Personnel and Productivity

In the crushing and screening section, four men work day and afternoon shifts 5 days a week - 10 shifts, or 40 man-shifts a week. Approximately 70 tons is produced per man-shift, including fine shale and dust, which amount to nearly 11 percent of the gross tonnage treated.

The net working expense of crushing and screening is approximately 34 cents a ton. The cost of the net shale (free of fines and dust) delivered to the crushers is \$4.67 a ton after accounting for 11 percent discarded as fines and dust. Therefore, the cost of shale delivered to the retorts is approximately \$5 a ton.

#### Retorting Section

This section of the plant receives shale from the storage bins, retorts it, disposes of the ash, and delivers oil vapors and gas to the treating section. Briefly, the present form of the plant was arrived at in the following manner:

In 1938, draft plans were prepared for the erection at Glen Davis of two tunnel-type retort ovens, each of 336 tons-a-day capacity, similar to a type used in Estonia and believed to offer some advantages over the retorts already tried in Australia. In March 1939, it was decided that such a plant would cost more to install than was available for this purpose, and that 64 retorts similar to those already erected at Newnes would be constructed.

Because of the urgent need for the early completion of the plant and production of gasoline - largely the result of the ominous international outlook - it was decided to salvage, from Newnes and elsewhere, retort materials that in other circumstances probably would not have been used.

The principle of side vapor-offtakes, which had been applied to some of the Newnes retorts, was disregarded in building this bench of retorts, and internal center oftakes were installed. The central vapor-offtakes did not prove satisfactory,

and although modifications made it possible to continue producing oil, major reconstruction was required to cure the trouble and attain the desired production. A second bench of 44 retorts, having four side offtakes for vapor removal, was constructed.

The original condensing plant also was quite incapable of efficiently handling the oil vapors and gases from the first bench, and financial arrangements were made to install a plant large enough to handle the expected production from both retort benches.

The whole of the reconstruction work was carried out under wartime conditions, and more than 2 years elapsed between placing of the first order and delivery of the materials. During this time, experiments were carried out on a house of four retorts fitted with improvised side offtakes. Data collected from these tests gave promise of greatly increased oil production when all retorts would be converted to this type. The old center offtake bench was closed down at the beginning of December 1946 following starting of the new bench and new product plant, and the work of converting the former bench to the new design was begun.

A brief description of the new retorting plant (fig. 10) follows: Shale is withdrawn from the storage bins by means of reciprocating plate feeders that deliver to a rubber belt conveyor driven by a 10 brake-horsepower electric motor, which, in turn, delivers to a second belt conveyor No. 11, which carries shale to the top of the retort bench (fig. 14). This latter conveyor is driven by a 10 brake-horsepower electric motor and is fitted with a Blake Denison weighing machine, which records the weight of the material carried by the belt.

Having reached the retort top, shale is fed from the belt conveyor into a steel-lip bucket conveyor capable of discharging its load at any point along its travel. The conveyor buckets are discharged by moving a tripping device to the selected point, which is over any one of a number of shale bunkers, and while the tripper remains at that point, all buckets are caused to swivel on their pins and tip enough to discharge their contents into the bunkers immediately below the tripper.

This conveyor is used also to transport ash from the residue elevators and delivers to the small ash-receiving bunkers, from whence the residue is used to start an empty retort. The retorts in their present form consist of two benches or batteries, one comprising 44 retorts and the other 60 retorts, four of the original 64 having been demolished in the course of reconstruction.

### Fell Retorts

The retorts are built in nests of "houses" of four retorts forming two lines or rows of retorts 5 feet from center to center. Each retort (fig. 15) consists of a vertical firebrick tube of circular cross section, 35 feet 6 inches tall, tapering from 3 feet inside diameter at the bottom to 2 feet diameter at the top and built from especially shaped 4-inch firebricks, provided at every third course with outward-jutting "butt bricks", which, in turn, rest against inward-jutting butt bricks built into the flue linings, thus providing lateral support while allowing vertical expansion.

The flue linings could be likened to another circular, 4-1/2-inch, brickwork structure built around the retort tube and separated from it by a 5-inch annular space in which the heating gas is burned. This annular space is divided into seven chambers by built-in cover courses, which close off the upward passage of the gas, except in one place, where a port communicates with the chamber above.



Figure 14. - Showing No. 11 conveyor from storage bins, lower right, to retorts.





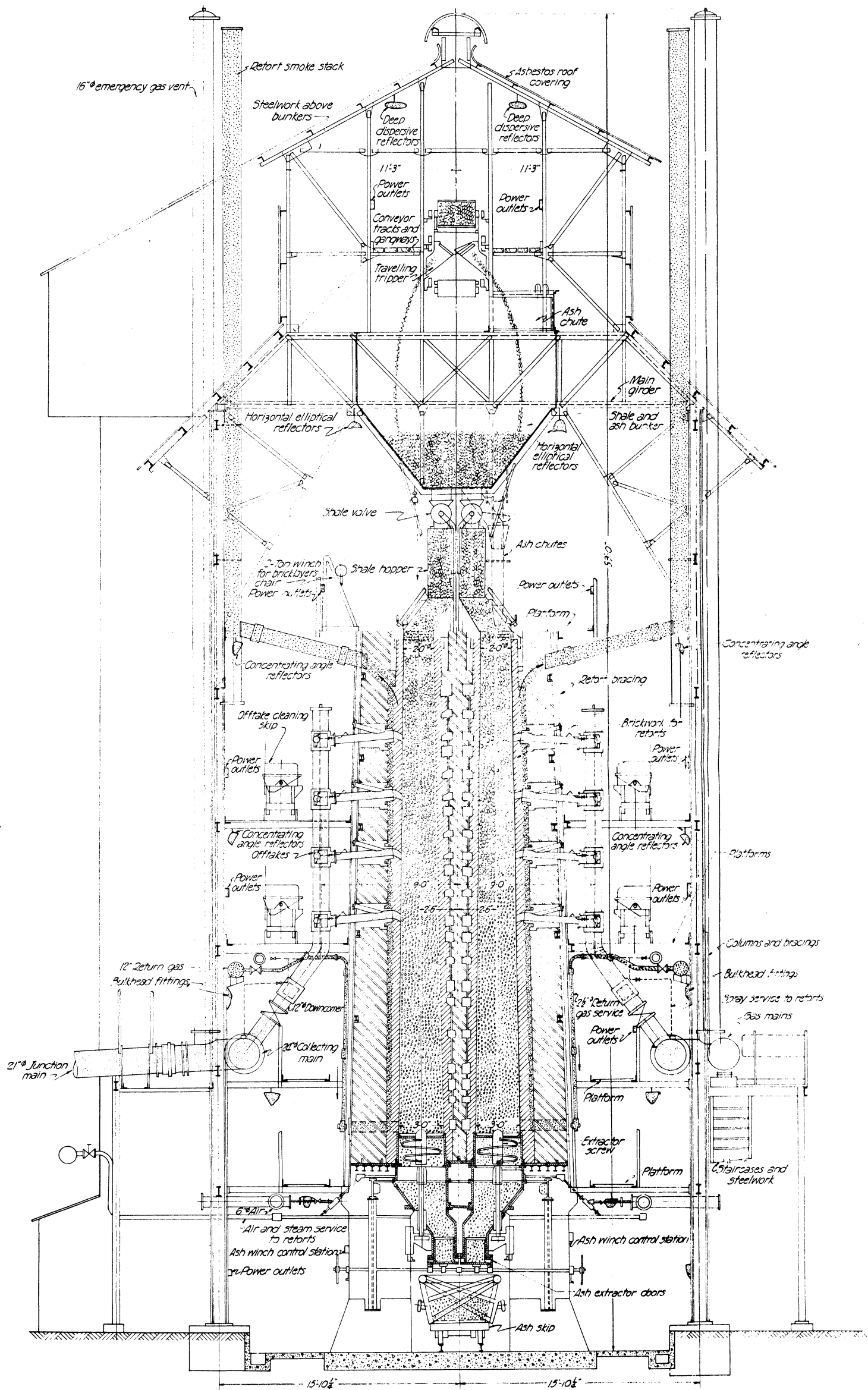


Figure 15. - Cross section of Fell retorts, No. 2 bench, Glen Davis, N. S. W., Australia.



A stop-wall divides the annular flue space from top to bottom of the retorts, and as the ports through the cover course alternate on each side of this stop-wall, the effect on the heating gas is to cause it to circle the retort tube clockwise, then counterclockwise alternately through the seven heating chambers, from the last of which the products of combustion escape into a chimney stack and thence to the atmosphere.

The flue linings are of common brick and are built outward to form a continuous straight structure encompassing the whole bench of retorts. Ports are provided through this common brickwork at intervals up each retort flue, so that the temperature of the retorts may be inspected. A 2-inch gas burner fitted with an air-control shutter at the base of the flue space supplies the necessary gas for heating. Temperatures are of the order of 1,800° F. at the base of the retorts, graduating to 800° F. in the upper chambers.

The retort tube is surmounted by a shale hopper fitted with a spigot-type charging valve immediately below the shale bunkers. The retort tube is designed so that when repairs are necessary a door can be removed and access gained for a man on a bosun's chair to inspect the brickwork.

Extractor screw. - The lower end of the retort tube is lined with a circular Meehanite<sup>20/</sup> housing to protect the brickwork from abrasion caused by an extractor screw 35 inches in diameter, upon which the spent shale rests. The extractor screw consists of three flight segments of Meehanite casting forming a helix of 405 degrees, or 1-1/8 turns, and having a lead of 11 inches at the periphery. This helix is driven by a 3-inch shaft supported directly beneath the screw by a spider bearing.

The upper segment of the extractor screw is keyed to the 3-inch shaft, whereas the lower two segments are fixed to a sleeve that passes over the shaft and extends downwards to a point immediately above the gear-box driving mechanism, where it terminates in a square nut affixed to the internal shaft by a removable pin. The effect of this mechanism is to allow the top segment of the screw to be rotated away from the other two when the affixing pin has been removed, thus allowing a tool to be passed through the opening to clean away scurf deposits adhering to the internal faces of the bricks.

A strengthened Glover-West gear box is mounted on the lower end of each extractor-screw shaft, consisting of a worm and worm-wheel drive of 11 to 1 reduction, which, in turn, is fitted with a ratchet wheel and suitable pawls actuated by two reciprocating bars, one on each side of the bench, which by their motion drive all the gear boxes. As the reciprocating bar moves, the extractor screws are caused to turn slightly with each stroke, thus dropping a portion of the spent shale into the spent-shale hoppers, one of which is attached to each retort. The rate of movement is adjustable by setting a shroud on the ratchet wheels at the required position.

As residue is discharged into the residue hoppers, the whole column of shale within the retort moves downward, leaving a corresponding space in the shale hopper surmounting the retort. At approximately 2-hour intervals, the charging valve is opened and the hopper is refilled from the shale bunker.

The spent shale accumulates in the spent-shale hopper, where it is quenched by a water spray. At hour intervals the spent shale is discharged into ash cars by opening ash-extractor doors, comprising a slide fitted with a rack and pinion and manually operated.

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<sup>20/</sup> A special cast iron, resistant to corrosion, abrasion, and high temperatures.

Steam at 13 pounds per square inch pressure is passed through a jet into each spent-shale hopper at the rate of 213 pounds an hour (74 gallons per ton of shale), and in its passage it acts as an injector for about 1,350 cubic feet of air an hour (approximately 4,000 cubic feet per ton). A pressure of about 2 inches water is thus set up in the spent-shale hopper.

Vapor offtakes and gas mains. - The side of each retort is fitted with four vapor-offtake ports formed by building special firebrick blocks into the retort tube walls, the lowest of which is 16 feet 4 inches from the retort base above the spent shale hopper, the second 4 feet 2 inches higher, the third 3 feet 6 inches above the second, and the fourth 4 feet 3 inches above the third.

Into each special brick a 6-inch Meehanite pipe is fitted, which projects from the face of the common brick and is coupled with a 12-inch vertical manifold pipe (downcomer), the latter acting as a collector for all four offtakes. These vertical manifolds are, in turn, connected to a 24-inch collecting main, which leads to the base of the primary cooler tower.

The whole of this gas-main system is held under partial vacuum, controlled to minus 1/2-inch water gage by an Askania regulator. This partial vacuum is held on the whole of the shale charge in the retorts down to the point at which the steam and injected air counteract the vacuum. Below this point, a pressure is built up gradually to 2 inches water in the region of the spent-shale hopper. These conditions appear to be the best compromise that can be obtained in balancing pressures inside and outside the retort and still permit the passage of the large volumes of air, steam, and gas required for the retorting operation.

Figure 16 is a side view of retort bench 2. The downcomers from each retort are barely visible in line with and below the waste-gas stacks. The gas- and oil-collecting main from the retorts (right) leads to the condensers (left). Figure 17 shows, from left to right, the spent-shale bunker and elevator, the retorts, the raw-shale elevator in the corrugated-iron housing, the collecting main in front of the elevator housing, and below, parallel to the line of retorts, the low-pressure steam main.

### Retort Operation

To start operation, a retort is brought up to full working temperature, with the offtake valves closed. Spent shale is charged through the base of the shale hopper by means of a light, movable chute, until the retort is filled to the region of the top takeoff, the remainder of the space being filled with raw shale, and the extractor screw is started slowly. Steam only is then injected.

As the column of shale moves downward, raw shale is charged at the rate of 8 to 9 tons per retort per day, and when a pressure becomes evident owing to distillation taking place, the valve of the top offtake is opened and then each successively downward until all four offtakes are open. Air is then added to the steam injected into the residue hopper, and the retort is working normally. During operation, a hard deposit known as "scurf" forms on the inside surface of the retort brickwork, being heaviest in the region of the bottom offtake, so that at approximately 25-day intervals each retort is emptied, the extractor screw opened, and the scurf deposit cleaned off with long poking bars. The scurf deposit usually is 3 to 4 inches thick at the bottom offtake, tapering off to disappear about 25 feet above the bottom of the retort.

The residue, after being discharged from the retort residue hoppers, is disposed of in the following manner: Four ore cars of the side-discharge type are connected to

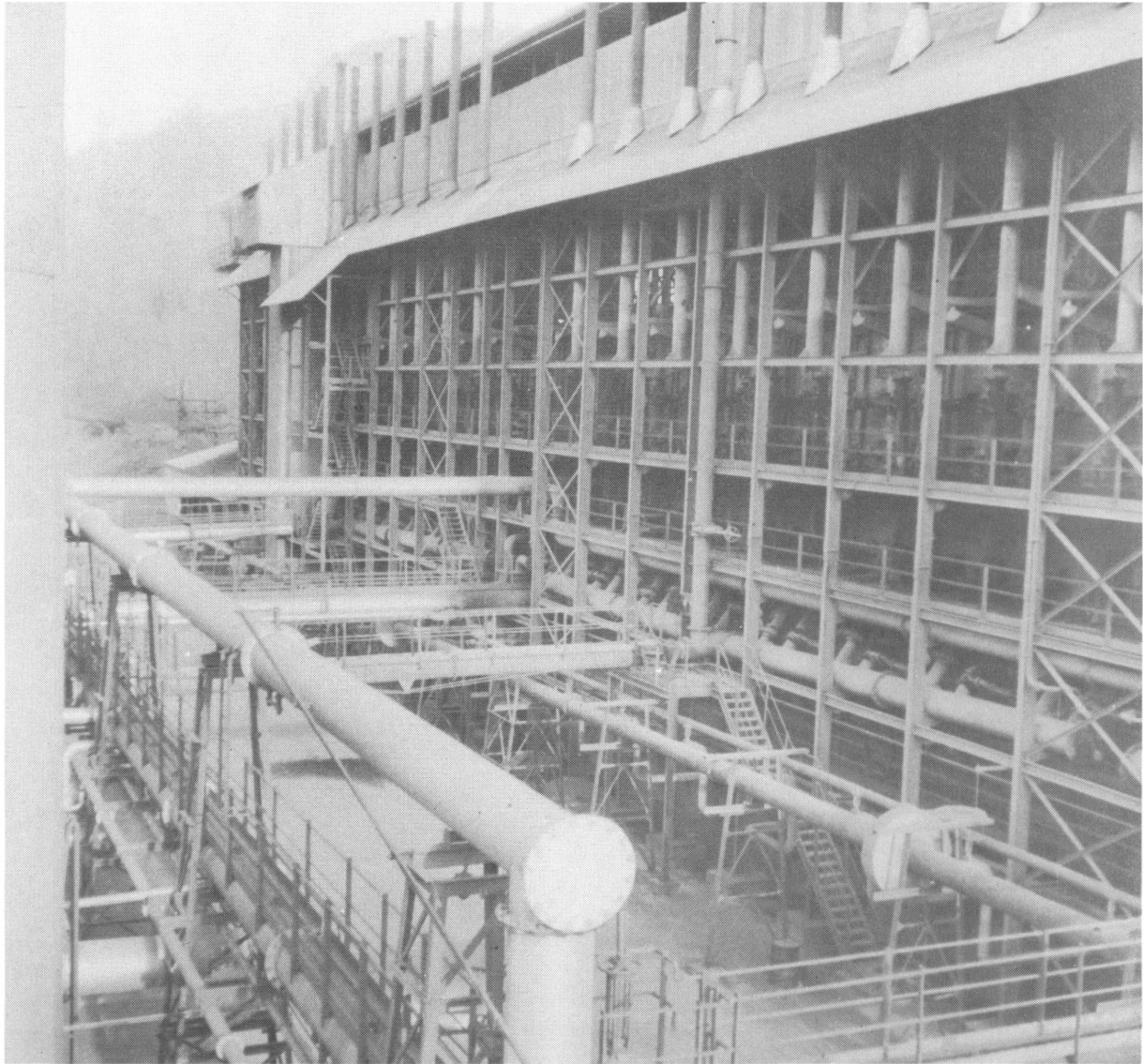


Figure 16. - Side view of bench 2, Fell retorts, Glen Davis, N. S. W., Australia. "Downcomers" from individual retorts barely visible in line with and below the waste-gas stacks. Gas and oil mains from retorts (right) to condensers, etc. (left).



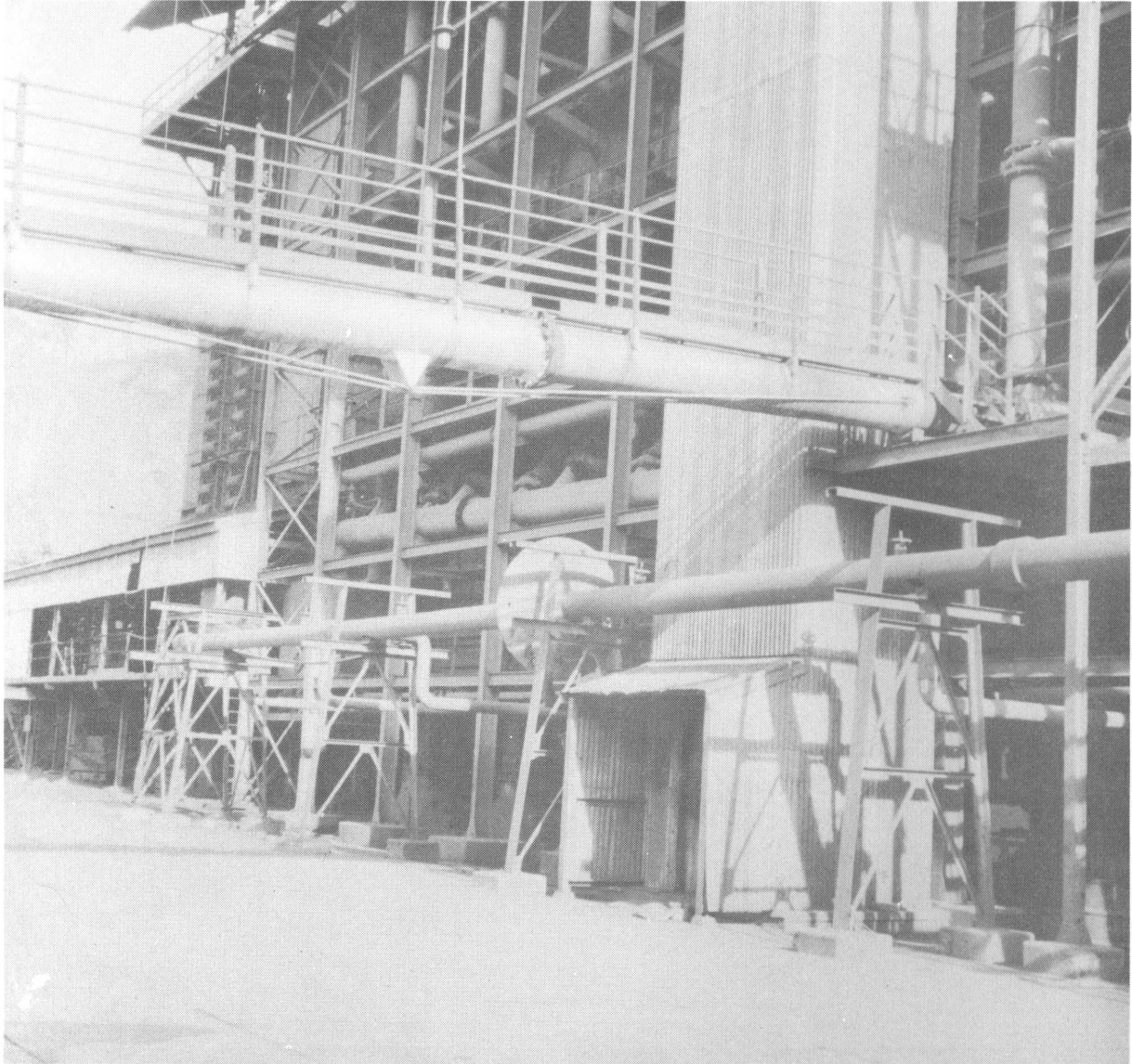


Figure 17. - Fell retorts, Glen Davis, N. S. W., Australia, showing, left to right, spent-shale bunkers and elevators, retorts, raw-shale elevator, collecting main, and, lower, low-pressure steam main.





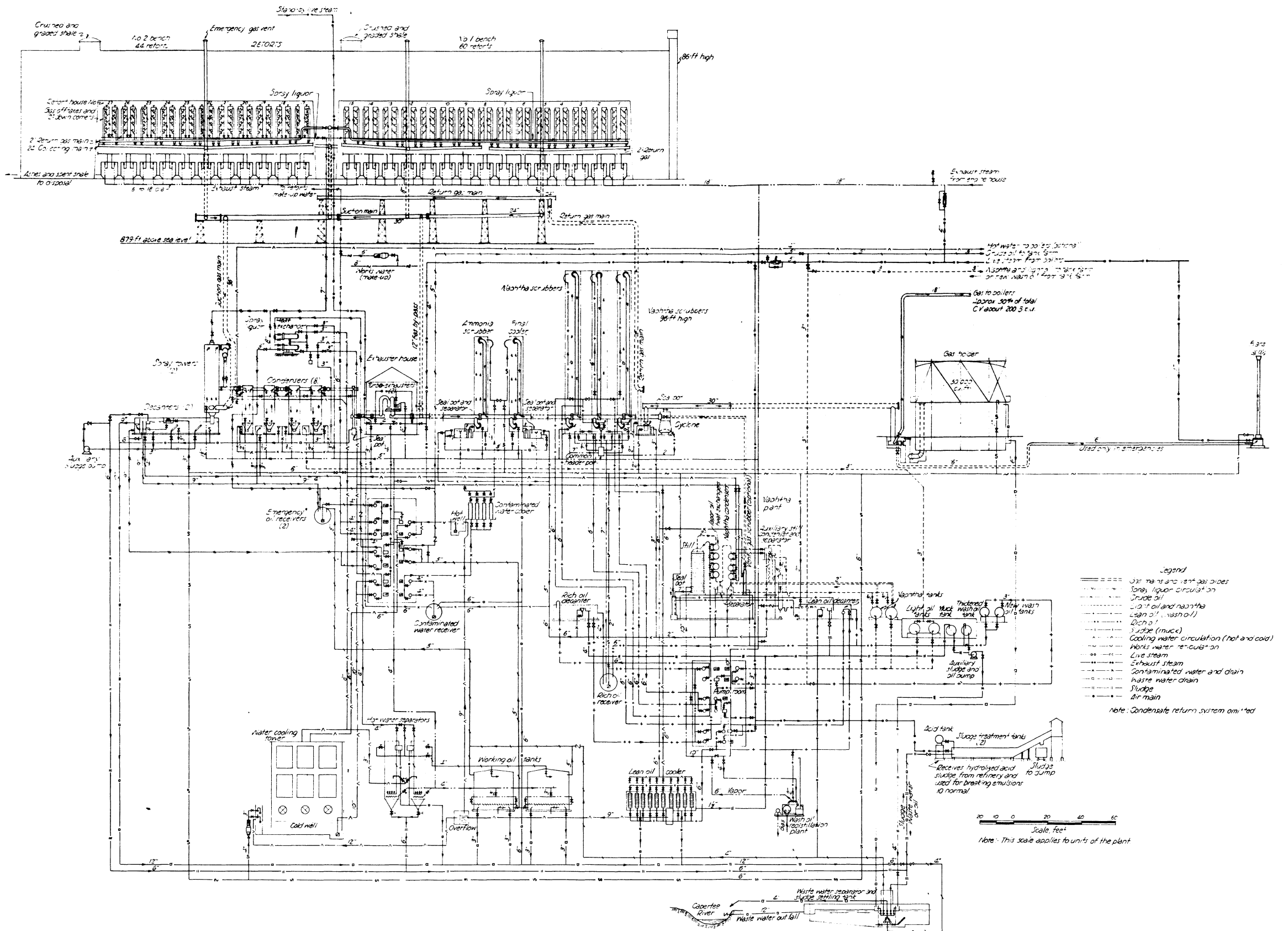


Figure 18. - Flow diagram of condensing and scrubbing plant of National Oil Proprietary, Ltd., Glen Davis, N. S. W., Australia.  
 After: (Woodall-Duckham (A/Asia) Pty. Ltd., Melbourne, Australia. Drawing 4006/D/E, dated December 3, 1945.)



an endless rope haulage, and their movement is controlled electrically from every third house of retorts. Each car has a door on each side that is hinged through suitable rigid connections (fig. 15) to the opposite side and is free to be lifted almost vertically, thereby allowing the contents to discharge. A projecting roller is fitted to the center of each door, and as the cars are drawn to the discharge point the rollers strike an automatic door-opening cam (fig. 10), the doors are thus lifted, and the residue discharges into a receiving hopper below rail level.

Having discharged, the leading car strikes a stop switch, which also shows a light on each control box, and the haulage stops. When the light appears, the operator presses the recall button and the cars return to the operator's position along the bench and are ready for refilling.

At its lower end the ash-receiving hopper is fitted with two chutes, each of which delivers to a lip-bucket elevator. These elevators encircle two residue bunkers, the tops of which are level with the top of the retort bench, and at their discharge point a chute arrangement allows residue to reach either the bunkers or the rear end of the shale-charging conveyor. The latter flow is used when retorts are recharged, as mentioned previously.

Residue accumulates in the residue bunkers (which have a capacity of 12,150 cubic feet) until it is removed in aerial ropeway buckets, which are filled on a loading station at the base of the bins. The ropeway buckets are borne on a bicable system and are tipped automatically by a movable tipping frame suspended at a pre-selected point between two towers (fig. 10). The empty buckets pass around a wheel at the return station and are unclipped automatically from the driving rope at the loading station. As an alternative, residue may be loaded into motor trucks direct from the bunkers.

#### Condensing and Treating Section

As the gases and vapors are drawn from the retorts through the vapor oftakes and collector mains, they pass through a primary ammonia-liquor spray in each off-take pipe ("spray service to retorts", fig. 15), which quenches the oil vapors and washes down tarry matter and ash that otherwise would deposit in the mains. An additional spray is fitted to the top of each 12-inch vertical manifold pipe, and others are set at intervals along the collecting mains.

The temperature of the spray liquor is controlled to 160° F. by passing the flow through a tubular heat exchanger ("spray liquor heat exchanger", fig. 18), to minimize the formation of emulsion, it having been found in practice that this temperature is above the dew-point of the water vapor in the gas. The spray liquor and condensate, together with solid particles, wash down the floor of the mains to the base of the primary cooler, where the liquor joins the runoff from the cooler sprays and enters one of the two crude-oil decanters (fig. 18). In these decanters the liquors are separated into crude oil, sludge emulsion, ammonia liquor, and heavy solid matter, in that order downwards. The ammonia liquor is then pumped through the heat exchanger and thus back to the sprays.

The crude oil is pumped to the base of the hot-water separator, where it is distributed into small streams, which pass upward through 10 feet of hot water, being washed in the process to remove residual solid matter and emulsions. From the top of this separator, the oil is gravitated to working-oil tanks of 72,000 gallons capacity, and, after being tested for dryness, is pumped to one of two 1,200,000-gallon crude-oil storage tanks, from which it is removed as necessary to serve as charge stock to the refinery.

Figure 19 is a photograph of a portion of the condensing and treating section showing, left to right, the wood-framed water-cooling tower, the hot-water separators, working oil tanks, and the lean-oil rack cooler shown at the bottom of figure 18. Figure 20 shows the naphtha scrubber behind the rack cooler and the Wilputte naphtha still and heat exchangers that constitute the naphtha plant (center of fig. 18). The 50,000-cubic-foot gas-holder is seen in the background and the end of the sludge-treatment equipment at the right of the picture.

Sludge from the decanters is passed to an acid-treating plant, where its temperature is raised to 212° F. by means of steam applied through jets. Dilute sulfuric acid is then added equivalent to 0.33 percent by volume of 100-percent acid, and the components of the sludge are resolved into oil, water, and solids. The oil is decanted, the water is run off to waste, and the solids remain in the treatment tank, from which they are removed periodically by a dragline scraper, which draws the solids up an incline and discharges into motor trucks. The solid material that is not removed in this manner is removed at intervals by switching to a duplicate decanter and draining the fouled unit to the waste-water separator, and solid material then is removed by opening access doors and cleaning by hand.

Gas and uncondensed vapors continue from the top of the primary cooler surmounting the decanter and enter a series of multitubular condensers in which cold water is circulated around the tubes, lowering the gas temperature to 70° F. at the point of leaving the last of the series, and resulting in further condensation of vapors. The cooling water is circulated over a timber-packed cooling tower equipped with six fans, which apply forced draft to the tower, the cool water being circulated to various coolers and condensers through the plant.

The whole of the gas-main system to this point is under partial vacuum, which is maintained by one of two exhausters driven by 80-horsepower turbines. Each unit is capable of handling 450,000 cubic feet of gas an hour at 2,900 revolutions per minute. The gas normally enters the exhauster at 3 inches of water vacuum, leaves at 9 inches of water pressure, and is forced through a timber-packed ammonia scrubber.

Contaminated water collected from various condensing vessels and cooled in a rack cooler is sprayed into the top of the ammonia scrubber, thus further washing the gas, which then passes to a second timber-packed tower known as the "final cooler" (fig. 18). Here either partly contaminated or fresh water may be sprayed at will, the gas being subjected to a final washing and cooling before entering the naphtha scrubbers.

The naphtha-scrubbing towers consist of three timber-packed, vertical, cylindrical vessels 96 feet tall and 9 feet in diameter. Gas enters the base of the first tower, passes through the timber packing to the top, through the main to the base of the second tower, and so on through the third.

#### Scrubber Oil

Straw oil, known as "lean absorption oil", is passed into the top of the third tower, flows over the timber packing in counterflow to the gas, and, upon reaching the bottom, is pumped to the top of the second tower, and so on to the first. During its passage, the oil absorbs naphtha from the gas and is now known as "rich oil." This oil passes to a rich-oil receiver, is pumped to a decanter, where any water is settled out, and thence to a Wilputte steam still, where the naphtha is removed by distillation, condensed, and received in a working tank after separation from aqueous condensate.

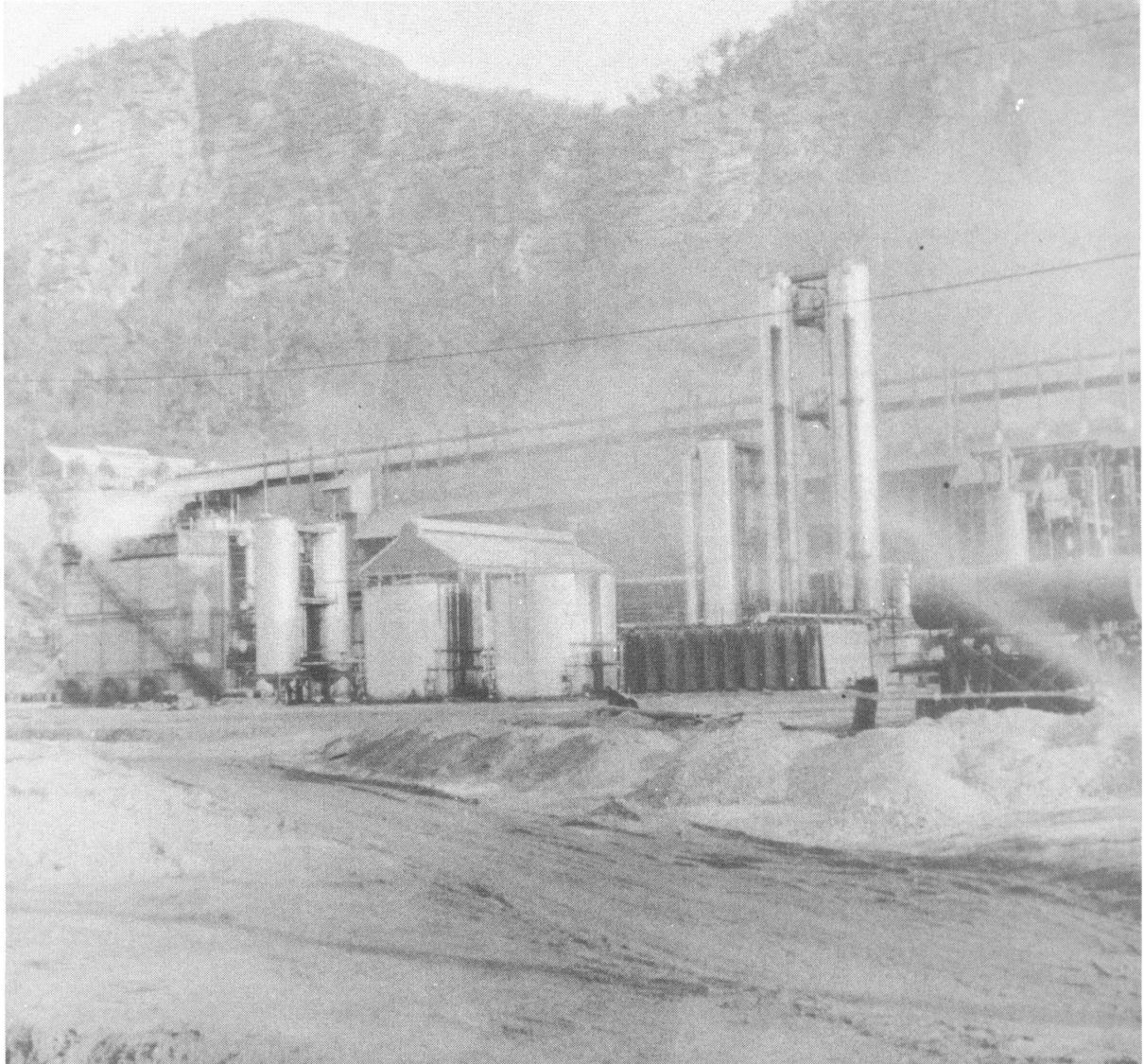


Figure 19. - Condensing and treating section, Glen Davis, N. S. W., Australia. Left to right cooling tower, hot-water separators, stock tanks, scrubber-oil cooler, sludge treatment equipment. Waste-gas stacks of retorts in background.



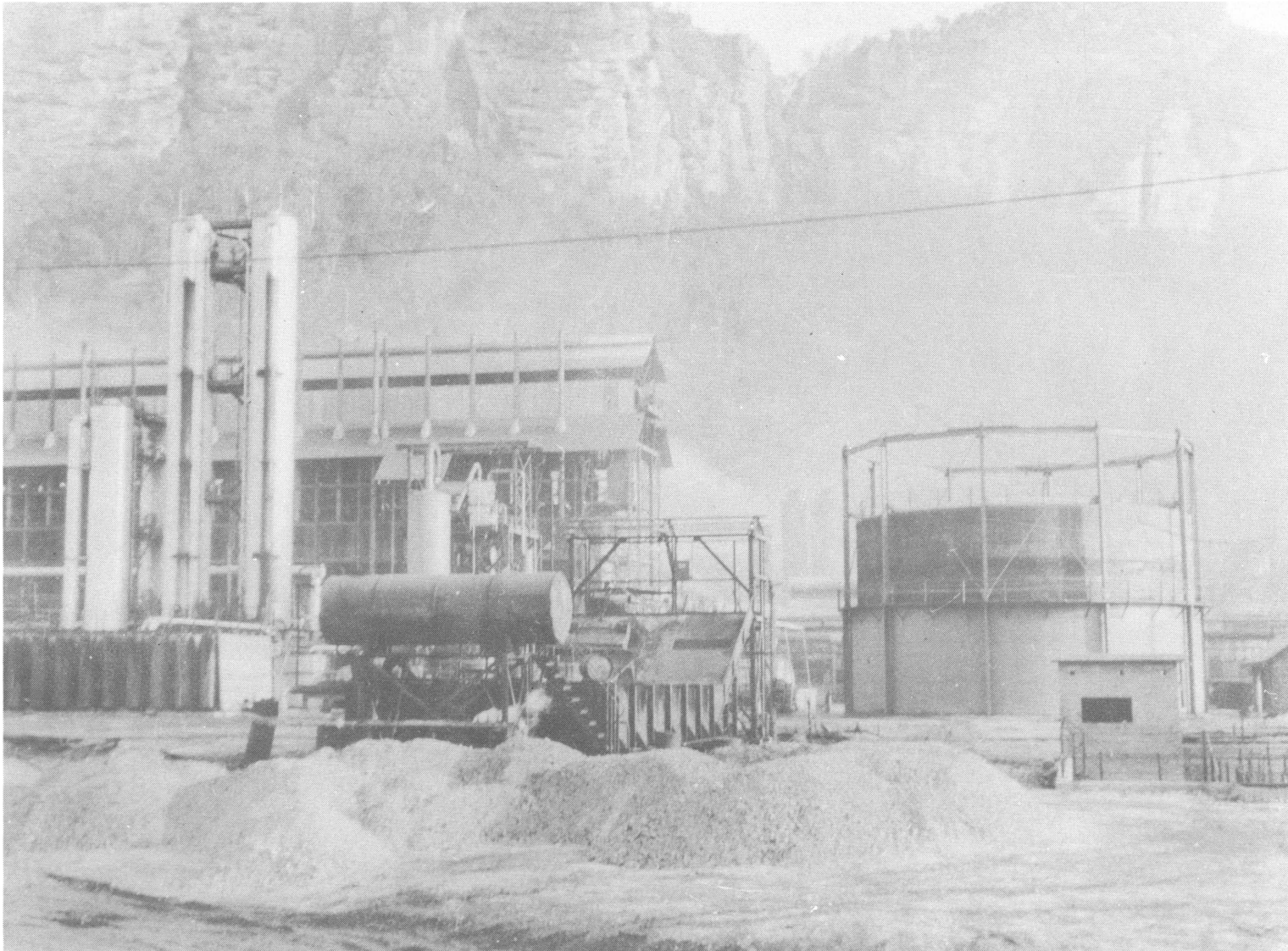


Figure 20. - Naphtha plant, Glen Davis, N. S. W., Australia. Left to right: Wilputte naphtha still, heat exchangers, etc., constitute the naphtha plant. Sludge-treatment equipment is seen at the right.





The hot scrubber oil is then passed through a decanter, cooled through a rack cooler to a temperature a few degrees above that of the gas in the scrubbers, and recirculated. Twelve centrifugal pumps are used in this process, five of which are steam turbine-driven.

#### Exhaust Steam and Gas

The exhaust steam from these five turbines joins the exhaust steam from other turbines in the plant and provides steam for distillation, retort injection, and for heating liquids.

The gas enters a cyclone after leaving the last of the scrubbers, where entrained scrubber oil is removed, and proceeds thence to a 50,000-cubic foot gas holder.

Approximately half of the gas from the holder is delivered to the retort burners, and most of the remainder to the power-house boilers. Any excess gas is burned in a flare.

#### Waste Water and Sludge

All waste water and sludge passes to a waste-water separator, where oil is skimmed from the surface and returned to an appropriate vessel, the water continuing on to waste.

#### Carbonizing Personnel and Productivity

The carbonizing department at the Glen Davis plant has approximately 100 employees, including 8 men in the crushing and screening section mentioned previously. The organization is approximately as follows:

1. Day men, working five 8-hour days a week:

- 1 Superintendent
- 1 Assistant superintendent
- 1 Clerk
- 1 Maintenance foreman
- 6 "Poking-down" men
- 2 Men on ash removal
- 4 Utility men

The superintendent, the assistant superintendent, and the clerk are rated as members of the staff.

2. The crushing and screening section requires eight men, who work two shifts a day, 5 days a week.

3. Each 8-hour shift has a shift foreman and two subforemen, who work 5 days a week. To operate a full week with employees who work 40 hours a week requires 4 men for each position and leaves a deficit of 8 hours. Therefore, there are 5 shift foremen and 9 subforemen to make up this deficit and to allow for absences.

4. Operation of the retorts requires 14 men on each shift, which accounts for 58.8 employees working 40 hours a week.

Absences among these employees are made up by substitutions, drawing on the subforemen and the day men.

Considering a week as a unit of operating time, the man-power requirements for the carbonizing department are:

	<u>Man-hours</u>
Staff.....	120
Foremen and subforemen.....	544
Day workers.....	480
Crushing and screening.....	320
Shift operators.....	<u>2,352</u>
Total per week.....	<u>3,816</u>

The total of 3,816 man-hours a week accounts for 95.4 employees working 40 hours a week. Absences for various causes require the maintenance of a force of approximately 100 men and some substitution of subforemen and day workers for shift operators.

During the first 6 months of 1947, the average throughput of the retorts was 422 tons of shale per calendar day. Based on a charging rate of 8.23 tons per retort per calendar day, this figure corresponds to an average of 51.3 retorts in operation. This low rate of utilization of the retorts probably was due mainly to the inability of the mine to provide enough shale, as more shale was retorted than was mined during that period; that is, a substantial quantity was withdrawn from storage. However, another reason for the low productivity is indicated by the "repairs and maintenance" cost, which was more than 45 percent as much as the "operating labor and supervision."

The principal item of cost for operating the retorts is steam, followed by operating labor and supervision and repairs and maintenance. These three items comprise more than 85 percent of the net working expense of the retorting plant.

The average assay value of the shale charged to the retorts was 67 gallons per ton, and the crude-oil production was 23,500 gallons a day, or 82.6 percent of the Gray-King assay value.

It will be noted that the assay value of the shale from the mine was 64 gallons a ton (p. 15), whereas the assay value of the shale as charged to the retorts was 67 gallons a ton. Disregarding differences due to errors in sampling and analysis, it seems likely that the increase in assay value between the mine and the retorts was due to the elimination of 11 percent of the shale as fines and dust in the crushing and screening operations. Fine shale and dust usually have a lower assay value than coarse shale from the same source, because the leaner shale is more friable.

Assay values of shales from individual beds and other chemical and physical properties of Australian shales are given later in this report under the section on Oil-Shale Research.

The net working expense for retorting shale, allowing credit for fuel gas supplied to the power-house boilers, was \$2.54 a ton. Adding the cost of the shale brings the total cost to \$7.37 a ton of shale or 13.3 cents a gallon of crude shale oil. These figures do not include return on investment, taxes, or profit.

#### Refining Department

The shale-oil refinery at Glen Davis in 1947 consisted essentially of the following major units:



Figure 21. - National Oil Proprietary, Ltd., oil-shale plant, Glen Davis, N. S. W., Australia. Refinery in background.



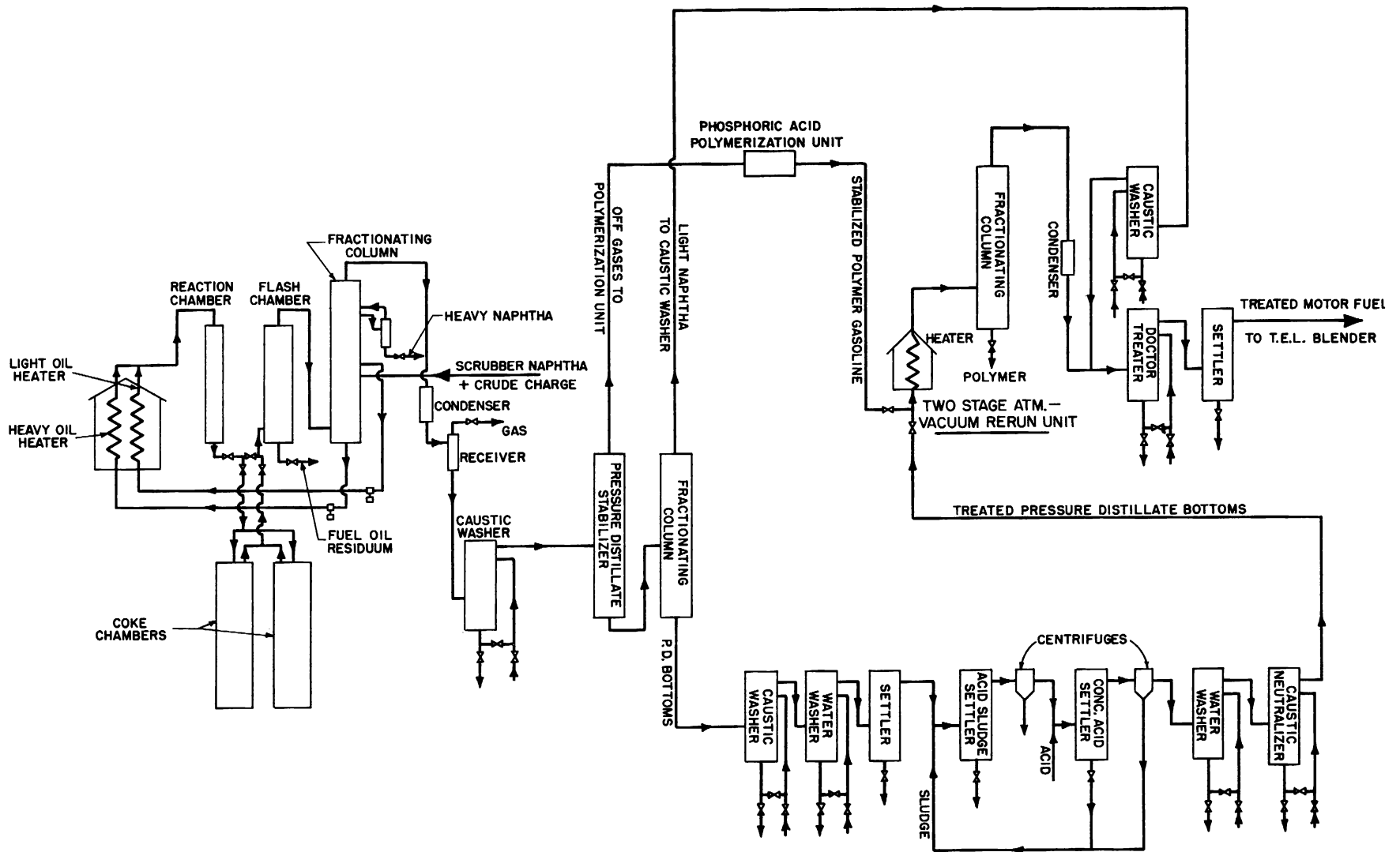


Figure 22. - Flow diagram of Glen Davis shale-oil refinery.



Two-coil selective Dubbs cracking unit  
Naphtha reforming unit  
Stabilizing unit  
High-pressure catalytic polymerization unit  
Continuous caustic and sulfuric acid treating units  
Atmospheric and vacuum re-run still  
Continuous doctor sweetening unit  
Tetraethyl lead blending unit  
Crude-oil and product storage.

The cracking plant (fig. 21) has a rated capacity of 1,600 barrels a day when running to residual fuel; it has run as high as 2,000 barrels a day by this type of operation. Its capacity when running to coke is 1,000 to 1,100 barrels a day. Both residual fuel and coking operations are used, the amount of crude run by each type of operation depending upon the plant needs for residual fuel and the market for coke. Normally, about equal quantities of crude are run to coke and to residual fuel.

Prior to September 1947, a blend of clean crude oil from the carbonization plant and the scrubber naphtha from the naphtha-recovery plant was charged to the main fractionating column of the cracking unit (fig. 22). The operating procedure described in this report is that used for the crude oil-naphtha charge. At the time of the authors' visit to the plant (Sept. 1947), arrangements had been completed to charge the scrubber naphtha separately to the reforming unit to increase its octane rating, thus excluding it from the cracking-unit charge. This change should have little effect on cracking operations, as about 90 percent of the naphtha is taken overhead from the fractionating tower and consequently is not cracked. The character of the stocks and products will be described following the discussion of plant operations.

#### Cracking Plant

The main fractionating column contains 16 bubble-tray decks and 8 side-to-side trays. The crude oil-naphtha charge is introduced into the column near the bottom of the bubble-deck section. Unstabilized gasoline, a mixture of cracked, straight-run, and scrubber naphthas, is taken overhead at a top temperature of 385° F. Formerly, a heavy naphtha was taken from the fifth tray when running to coke and recharged to the unit under residual-fuel operations. Later, this side cut was used as charging stock to the reforming unit. The naphtha was withdrawn for the purpose of purging, from the light oil circuit, the naphthalene, which tended to accumulate in that oil and depress its crackability. By so doing, it was found possible to raise the throughput of the unit to the design rating, 1,600 barrels a day, when cracking to coke. However, the naphtha, which was highly unsaturated and often contained as much as 1 percent of naphthalene, formed coke so freely in the reformer that the operation was discontinued.

Heavy-oil cracking stock, including recycle, is pumped from the bottom of the column at the rate of about 5,500 barrels a day at 400 pounds per square inch pressure into the heavy-oil section of the cracking heater. This section contains 82 30-foot tubes 3-1/2 inches outside diameter and 2-7/8 inches inside diameter, giving a flow length of 2,460 feet. The velocity of oil flow at 60° F. through the tubes is normally 7 to 8 feet a second. The oil is discharged into the reaction chamber at about 310 pounds per square inch pressure at a transfer temperature of 925° to 930° F.

Light-oil cracking stock, including recycle, at the rate of 1,500 barrels a day is picked up between the 11th and 12th trays of the fractionating column. This oil has a boiling range of approximately 350° to 600° F.

The light-oil cracking section contains 100 30-foot tubes 3 inches outside diameter and 2-3/8 inches inside diameter, giving a flow length of 3,000 feet. The light-oil fraction from the column is pumped through this section at a rate of 5 feet a second, a pressure of 600 pounds per square inch, and transferred at 1,005° to 1,025° F. to the reaction chamber with a pressure reduction to 310 pounds.

The pressure of the oil from the reaction chamber is reduced to 125 pounds per square inch, and its temperature is reduced by quenching before admission to the flash chamber. The vapors from the flash chamber go to the fractionating column previously mentioned, and the residual bottoms to fuel oil storage in residuum-type cracking operations.

A yield of 30 to 33 percent of residual fuel is usual. This fuel oil is not marketed but is used as plant fuel. It has a specific gravity of 1.0+, a flash point of 130° F., and a sulfur content of about 1 percent.

In the non-residuum type of operation, two coke chambers (10 ft. x 40 ft.) are used alternately. Each chamber is on stream about 36 hours before cleanout is necessary. The coiled-cable method is used for coke removal. This type of operation yields approximately 25 percent coke based on the crude oil charge and an estimated 14 percent gas. The coke is of good quality, usually containing less than 10 percent volatiles and less than 0.1 percent ash. It finds a good market for carbide manufacture.

The heavy and light oils from the fractionating column are recycled to the respective cracking coils to an ultimate yield of gas, gasoline, and coke, or, alternately, gas, gasoline, and residual fuel oil.

A new reforming unit with a capacity of 350 barrels a day was completed in 1947. It was to charge a sidestream of naphtha for improving its octane number. However, as mentioned previously, when charging the naphtha sidestream it was found that temperatures above 1,000° F. caused much coke formation which plugged the tubes rapidly. By holding the transfer temperature at 1,000° F. or below, at 600 pounds per square-inch pressure and the cross-over temperature at 850° F., the unit could be operated satisfactorily, but under these conditions very little reforming was effected. Plans were completed in September 1947 to charge the scrubber naphtha (cut to 400° F. end point) from the carbonization plant directly to the reformer.

The overhead from the main fractionating column is cooled and separated, and the condensate is washed with caustic soda, primarily for the removal of tar acids, in a continuous countercurrent column. The pressure distillate so washed is stabilized to 5.5 pounds per square inch vapor pressure in a conventional stabilizing unit. The stabilized pressure distillate is refractionated into two fractions for further treatment, one consisting of about 30 percent light naphtha having an end point of 250° F. and the second of 70 percent bottoms.

#### Polymerization Unit

The off-gases from the pressure distillate stabilizer are washed with caustic soda, then fed directly to the polymerization unit. This unit is a Universal Oil Products Co. high-pressure phosphoric acid catalyst type. It contains three catalyst chambers and operates at a pressure of 1,100 pounds per square inch at 425° F. A yield of 4 to 4.5 percent polymer is obtained, based on the cracking plant charge. The polymer has a clear octane rating of 85 A.S.T.M. motor method and a blending value of 100 octane. The polymer is stabilized to 24-pounds vapor pressure, retaining most of the butanes. A small quantity (240 gallons a day) of butane is being



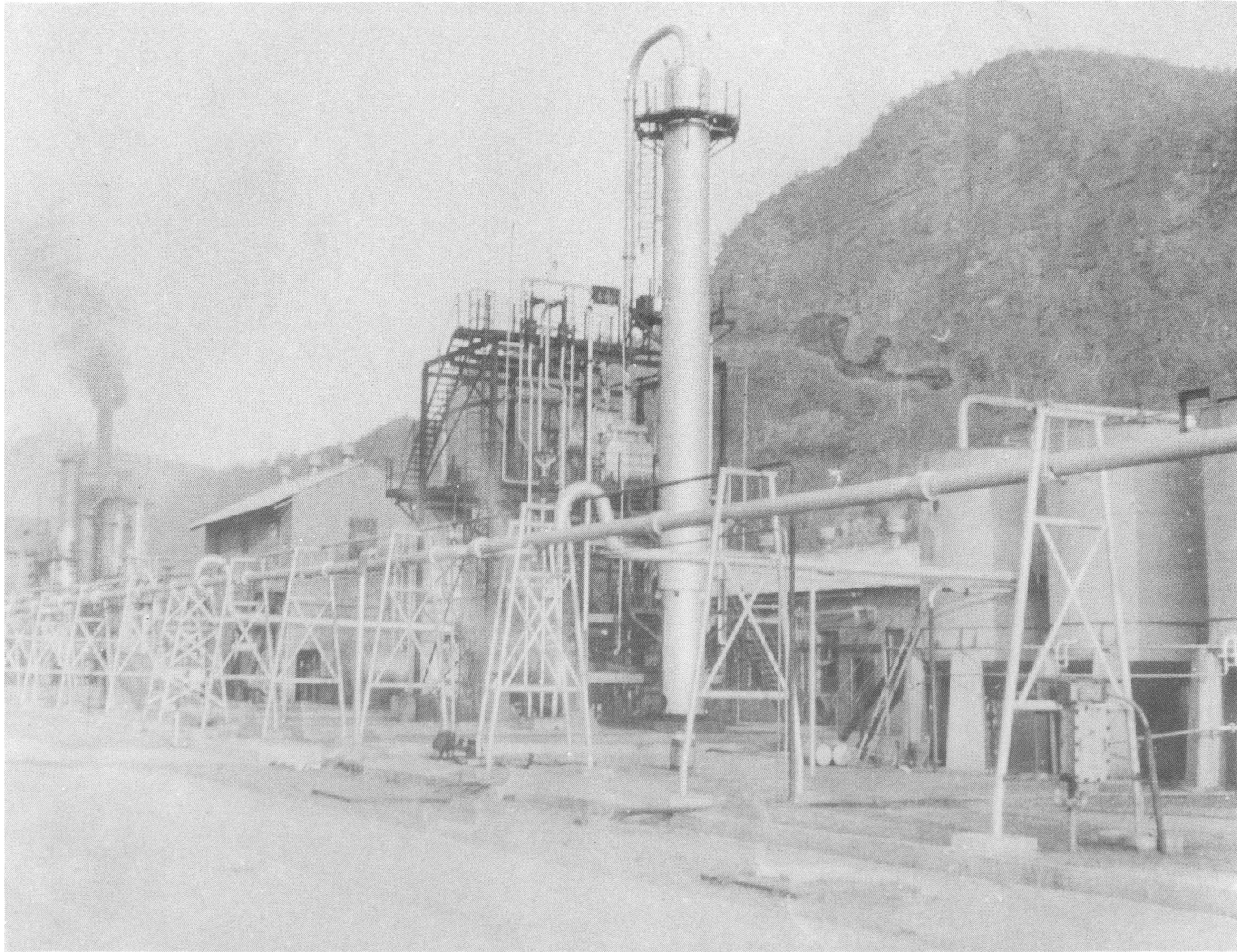


Figure 23. - Glen Davis shale-oil refinery. Rerun unit in the center and dubbs cracking unit in left background.



recovered from the off gases, and plans were underway to recover a propane-butane mixture for the liquefied petroleum gas market.

#### Treating

The light naphtha is caustic washed only before blending to produce a final motor fuel for doctor treatment. The pressure distillate bottoms are caustic washed to remove residual traces of tar acids and volatile mercaptans and then washed with water. After settling, the bottoms are treated continuously:

1. With once-used acid sludge at atmospheric temperature - contact time approximately 3 minutes;
2. By centrifugal separation of sludge;
3. With 3 to 7 pounds per barrel of 98 percent sulfuric acid - the quantity of acid depending on the sulfur content of the distillate - with a contact time of approximately 2 minutes;
4. By centrifugal separation of sludge;
5. By water washing; and
6. By caustic washing.

The tar bases are removed by the sludge and acid treatment; the sulfur content, ranging from 0.6 to 0.3 percent, is reduced to below 0.2 percent; and the gums and gum-forming constituents are reduced by solution and polymerization. Mapstone<sup>21/</sup> has given a more detailed description of treating procedure and the various problems involved.

The acid-treated pressure-distillate bottoms and the stabilized polymer are mixed and redistilled in a 2-stage atmospheric and vacuum still to about 385° F. end point. The heavy ends are used for plant fuel. The distilled gasoline is blended with the 30 percent light naphtha which has been caustic-washed, and the resulting blend is doctor-treated continuously. The gasoline is finally inhibited with 0.005 percent Universal Oil Products Co. No. 4 inhibitor and sent to storage. Tetraethyl-lead, up to 1.25 ml. a gallon, is blended with the gasoline in storage to produce a finished motor fuel with a vapor pressure of 8 pounds per square inch and an A.S.T.M. Motor Octane Number of 70. The octane number of the unleaded motor fuel usually ranges from 63 to 65.

About 5 to 10 barrels a day of mixed cresols are recovered from the caustic solution after washing the pressure distillate. The cresols are separated from solution by bubbling combustion gases containing carbon dioxide through the solution. The acid sludge from refining operations is hydrolyzed, and the weak acid is used to break emulsions in the carbonization plant.

#### Distribution of Products

Because Glen Davis has no rail connection, the finished motor fuel is pumped by pipeline to the most convenient point on the Main Western Railway, which is at Newnes

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<sup>21/</sup> Mapstone, G. E., Shale-Oil Emulsions and Sludges: Petr. Ref., vol. 24, No. 6, June 1945, pp. 209-17.

Junction, a distance of 30 miles. The pipeline is 3 inches in internal diameter, and the working pressure is 1,550 pounds per square inch. A Wilson-Snyder duplex plunger pump at Glen Davis provides the necessary force to deliver the product to the railway pipeline terminal, which is at an elevation of 2,500 feet above the refinery. From this point the motor fuel is distributed to customers by Pool Petroleum Proprietary, Ltd. The motor fuel is sold to Pool Petroleum Proprietary at the landed cost, duty paid, of imported gasoline of similar quality at Sydney. National Oil Proprietary pays the excise (duty) and therefore gets full price for its product.

#### Operating Schedule

The refinery at Glen Davis operates intermittently, as its capacity exceeds the output of the carbonization plant, the output of which, in turn, has been limited by the production of the mine. During the first 6 months of 1947, the refinery was on stream 70 days and off stream 111 days. During the 70 days, slightly more than 88,500 barrels of crude oil was processed, an average of 1,265 barrels a day. The plant usually operates 30 to 40 days between shutdowns. However, the last operation prior to September 1947 was for 52 days. Normally, a backlog of about 30,000 barrels of crude oil is accumulated before a refinery operating period is started.

#### Refining Utilities

During refining operations, hourly electrical requirements are 10 kw. for the cracking plant and 22 kw. for the treating plant. Hourly steam requirements are 12,000 pounds for cracking operations and 6,000 pounds for treating.

#### Refinery Personnel

Operating personnel in the cracking plant are four men per shift and in the treating plant three men per shift. In addition, two tank gagers and one instrument man serve the entire refinery. Maintenance personnel consists of one pipefitter and one helper for each shift. The de-coking crew consists of 11 men when at full strength.

#### Refining Costs<sup>22/</sup>

##### Cracking and Polymerization

During the first 6 months of 1947, 88,543 barrels (3,718,805 gallons) of crude oil was processed to produce 50,967 barrels (2,140,614 gallons) of raw pressure distillate and polymers for a recovery of 57.6 percent. Operating costs, including labor and supervision, utilities, repair and maintenance, chemicals, royalty, and general works costs and administration were \$0.0273 per gallon of product. By far the largest single item of operating cost was steam, which amounted to \$0.0106 per gallon of product. Byproducts of cresols, coke, and propane-butane were credited at the rate of \$0.0191 per gallon of pressure distillate and polymer, leaving a net operating cost of \$0.0082 exclusive of the cost charged the refinery for crude oil and off-stream plant charges. Crude feed and off-stream charges, based on the quantity of pressure distillate and polymer product, were \$0.2261 and \$0.0156, respectively, making an over-all cost of \$0.25 per gallon of product, or \$0.148 per gallon of crude charged.

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<sup>22/</sup> Exclusive of depreciation and return on capital investment.

## Treating

During the same period, treating operating costs were \$0.017 per gallon of treated product, exclusive of feed and off-stream charges and credit for byproducts. Including a feed charge of \$0.2791, an off-stream charge of \$0.007, and crediting byproducts with \$0.0007 per gallon of treated products gives a net cost of treated gasoline of \$0.3028 a gallon. The largest items of operating expense for treating were steam at \$0.0061 and chemicals at \$0.0045 per gallon of product.

The over-all recovery of treated gasoline from pressure distillate and polymer was 89.57 percent.

The intermittent operation of the plant caused operating costs to be higher than they would be by continuous operation.

### Refinery Operating Experience

The operation of the Glen Davis shale-oil refinery has been satisfactory, considering the characteristics of the crude-oil feed stock, which contains relatively large quantities of nitrogen and sulfur compounds and a high percentage of olefins compared to petroleum crude oils.

Some difficulty has been experienced owing to corrosion in certain parts of the refining equipment, but a comprehensive study of the causes of the corrosion by Mapstone and McLaren<sup>23/</sup> developed preventive measures that eliminated further trouble. Their diagnosis of the problem is interesting and unique, in that a similar situation would be encountered only in distilling and condensing materials, such as shale oils, that contain nitrogen compounds or tar bases and would seldom, if ever, be encountered in petroleum refining.

Cracking the tar bases in the crude shale oil produces small quantities of hydrogen cyanide. In the presence of water and ammonia, part of the hydrogen cyanide is hydrolyzed to formic acid, the remainder combining with the iron products of corrosion to form prussian blue. The formic acid causes corrosion in the upper section of the pressure distillate stabilizer, whereas in other sections of the plant the presence of tar bases inhibits this form of corrosion. The installation of a countercurrent caustic-soda wash between the distillate receiver and the stabilizer effected satisfactory removal of the acidic corrosive material.

### Characteristics of Crude Oil and Products

Analyses of crude shale oils from the shales of Newnes and Capertee Valley (Glen Davis), New South Wales and Tasmanian shales, together with the properties of their distillation products were presented by Kurth and Rogers<sup>24/</sup> at the 1938 Oil-Shale and Cannel Coal Conference held in Scotland and the results published in the proceedings of the conference. Egloff, Morrell, and Zimmerman<sup>25/</sup> presented the results of pilot-plant cracking, distillation, and treating experiments on shale oil from the Newnes district, New South Wales, and on shale oils from other countries at the same conference.

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<sup>23/</sup> Mapstone, G. E., and McLaren, C. B., Corrosion Problems in Cracking Shale Oil: Pet. Ref., vol. 25, No. 2, Feb. 1946, pp. 49-52.

<sup>24/</sup> See footnote 4.

<sup>25/</sup> Egloff, Gustav, Morrell, J. C., and Zimmerman, G. B., Motor Fuels from Cracking Shale Oils. Oil Shale and Cannel Coal: Proceedings of a Conference held in Scotland, June 1938, The Institute of Petroleum, London, 1938, pp. 366-73.

The authors were given analyses of some stocks and products, and, in addition, samples of crude oil, scrubber naphtha, raw and treated pressure distillate, and finished motor fuel were furnished by National Oil Proprietary for overseas shipment through the American consulate at Sydney to the Bureau of Mines Petroleum and Oil-Shale Experiment Station, Laramie, Wyo. More than a year elapsed between collecting the samples and completing the analyses; consequently, some changes in the composition and properties of the products may have occurred. These changes would be due largely to polymerization, which formed gums, raised the distillation end points, and increased the color in the oils.

### Crude Oil

Table 2 gives a Bureau of Mines crude shale-oil analysis of a representative sample of crude oil as produced at the Glen Davis plant in the latter part of 1947. The crude oil does not contain the scrubber naphtha recovered from the gases resulting from retorting the shale.

TABLE\* 2. - Bureau of Mines crude shale-oil analysis

Source of shale: National Oil Proprietary, Ltd., Glen Davis, N.S.W., Australia.  
 Eduction method: Modified Pumpherton retorts.

#### SUMMARY OF PROPERTIES

<u>Properties of crude shale oil</u>		<u>Distillation summary</u>			
Specific gravity.....	0.893				
A.P.I. gravity.....	26.9	Naphtha (fractions 1-7)	14.6	0.782	49.4
Sulfur, percent.....	0.56	Light distillate			
Nitrogen, percent.....	0.52	(fractions 8-11).....	20.4	.846	35.8
Pour point, °F.....	60	Heavy distillate			
Viscosity, S.U.,		(fractions 12-15).....	37.1	.897	26.2
seconds at 100° F....	54	Residuum.....	27.2	.956	16.5
		Loss.....	0.7		

#### ANALYTICAL DATA

<u>Distillation at 760 mm. Hg. pressure</u>							<u>Naphtha composite (fractions 1-7)</u>	
Fraction No.	Cut at-		Per-cent	Sum, percent	Gravity		Percent	Percent
	°C.	°F.			Specific 60/60°F.	°A.P.I. 60°F.		
1.....	50	122					Tar acids.....	1.4
2.....	75	167					Tar bases.....	0
3.....	100	212					Neutral oil:	
4.....	125	257					Paraffins and	
5.....	150	302	2.3	2.3	0.753	56.4	naphthenes....	45
6.....	175	347	4.5	6.8	0.771	52.0	Olefins.....	43
7.....	200	392	7.8	14.6	0.796	46.3	Aromatics*.....	12

\*Including sulfur and nitrogen compounds

TABLE 2. - Bureau of Mines crude shale-oil analysis (Cont.)

Distillation at 40 mm. Hg pressure

Fraction No.	Cut at-		Per-cent	Sum percent	Gravity		Viscosity at 100°F.		Aniline point °C.	Cloud test °F.
	°C.	°F.			Specific 60/60°F.	A.P.I. 60°F.	K.V. centi-stokes	S.U. sec.		
8.....	125	257	1.6	16.2	0.809	43.4	1.9	32	46.2	5
9.....	150	302	5.8	22.2	0.836	37.8				
10.....	175	347	6.3	28.3	0.848	35.4	2.1	33	49.2	20
11.....	200	392	6.7	35.0	0.861	32.8	3.6	38	50.0	35
12.....	225	437	7.8	42.8	0.876	30.0	5.6	44		45
13.....	250	482	8.5	51.3	0.887	28.0	9.1	56		55
14.....	275	527	9.0	60.3	0.904	25.0	15.3	79		75
15.....	300	572	11.8	72.1	0.915	23.1	47.3	220		95
Residuum			27.2	99.3	0.956	16.5				

Carbon residue of residuum, 3.0 percent; carbon residue of crude, 1.1 percent. Ash on residuum, 0.2 percent; ash on crude, 0.07 percent.

The analysis shows the boiling-range distribution of the crude oil, occurrence of paraffin wax as indicated by the pour point of the crude and cloud temperatures of fractions, and low content of asphaltic constituents, as indicated by the low carbon residue of crude oil. The hydrocarbon-type analysis<sup>26/</sup> of the composite naphtha is indicative of the refining requirements and character of motor fuel that may be produced from it and, in a general way, the probable composition of higher-boiling fractions, which, with other properties such as aniline point, cloud temperature, gravity, and viscosity, indicates the suitability of these fractions for the manufacture of commercial products.

Great caution should be exercised in the use of characterization factors, gravity-boiling point relationships, etc., developed from petroleum refining experience, to indicate the refining potentialities of shale oils. These factors usually are misleading owing to the great differences in composition of crude petroleum and shale oils. The greatest differences are caused by the presence of large quantities of unsaturated hydrocarbons, oxygen, and nitrogen compounds, which greatly affect the response of the shale oils to petroleum-refining technique.

#### Scrubber Naphtha

Table 3 gives the results of a distillation and analysis of 4 percent fractions of scrubber naphtha made in the laboratories of National Oil Proprietary at Glen Davis in August 1947. A 2.5 liter charge of scrubber naphtha was distilled from a 2-gallon pot still through a 4-inch column of 1-inch pipe packed with a roll of fine wire gauze. Presumably, the mercaptan content of each fraction was determined by Mapstone's<sup>27/</sup> modification of the silver nitrate method of Borgstrom and Reid<sup>28/</sup> and requires the removal of tar bases before determination of the mercaptan.

<sup>26/</sup> Dinneen, G. U., Bailey, C. W., Smith, J. R., and Ball, John S., Shale-Oil Naphthas, Analysis of Small Samples by the Silica Gel Adsorption Method: Ind. Eng. Chem. (anal. ed.), vol. 19, No. 12, 1947, pp. 992-8.

<sup>27/</sup> Mapstone, Geo. E., The Determination of Mercaptans in Gasoline: Australian Chem. Inst. Jour. and Proc., vol. 13, June 1946, pp. 232-238.

<sup>28/</sup> Borgstrom, P., and Reid, E. E., Quantitative Determination of Mercaptans in Naphtha: Ind. Eng. Chem. (anal. ed.), vol. 1, Oct. 15, 1929, pp. 186.

TABLE 3. - Properties of fractions of scrubber naphtha from retort gases, Glen Davis, N.S.W., Australia

Fraction No.	Boiling range, °F.	Volume collected, ml.	Sp. gr., 60°/60°F.	Sulfur content		
				Total percent	Mercaptan, percent	Non-mercaptan, percent
1.....	70 -154	100	0.670	0.10	0.100	-
2.....	154 -166	106	.691	.11	.095	0.02
3.....	166 -173.5	105	.695	.12	.088	.03
4.....	173.5-179	102	.700	.16	.078	.08
5.....	179 -185	103	.703	.13	.071	.06
6.....	185 -190.5	103	.707	.16	.063	.10
7.....	190.5-196	103	.712	.11	.057	.05
8.....	196 -201	103	.716	.10	.052	.05
9.....	201 -207	102	.719	.14	.048	.09
10.....	207 -213	100	.722	.21	.046	.16
11.....	213 -219	102	.726	.20	.042	.16
12.....	219 -225	100	.729	.26	.040	.22
13.....	225 -240	103	.736	.25	.031	.22
14.....	240 -248	104	.741	.32	.028	.29
15.....	248 -257	101	.747	.33	.026	.30
16.....	257 -266	104	.752	.33	.026	.30
17.....	266 -278	102	.756	.37	.027	.34
18.....	278 -294	101	.762	.42	.028	.39
19.....	294 -311	101	.774	.44	.026	.41
20.....	311 -331	102	.786	.46	.026	.43
21.....	331 -353	101	.804	.54	.025 <sup>2/</sup>	.51
22.....	353 -382	102	.834	.73	.025 <sup>2/</sup>	.71
23.....	382 -410	100	.875	.72	.015 <sup>2/</sup>	.70
24.....	410 -444	100	.910	.76	.011 <sup>2/</sup>	.75
25.....	Total temp. 449	60	.935	-	.004 <sup>2/</sup>	-
	Bottoms <sup>3/</sup>	32	-	-	-	-
	Original					
	121 -460	-	.756	.35	.042	.31

1/ Analysis made by National Oil Proprietary, Ltd., laboratories, August 1947.

2/ H<sub>2</sub>S also present but removed before analysis.

3/ Heavy viscous oil.

The analysis of the sample of scrubber naphtha shipped to the Bureau and analyzed at Laramie is shown in table 4. This sample had a lower end point than that analyzed at Glen Davis; consequently, it has a lower sulfur content, as the components of higher sulfur content are in the heavy ends.

The octane number of the unleaded scrubber naphthas is 56.1, indicating that the plan of the National Oil Proprietary to reform the scrubber naphtha, if successful, should substantially increase the octane number of the final motor-fuel product.

#### Pressure Distillate

Before chemical treatment, the pressure distillate is fractionated, as previously described, into about 30 percent light naphtha and 70 percent heavy naphtha. The light naphtha is caustic-washed only, whereas the heavy naphtha is acid-treated as well. Table 5 gives analyses of the raw pressure distillate and treated light and heavy naphthas.



TABLE 4. - Properties of scrubber naphtha from retort gases,  
Glen Davis, N.S.W., Australia<sup>1/</sup>

Properties	Untreated naphtha
Sp. gr. 60°/60°F. ....	0.732
Degrees A.P.I. ....	61.8
Tar bases, volume percent.....	.6
Tar acids, volume percent.....	.5
Sulfur, weight percent.....	.20
Nitrogen, weight percent.....	.11
Hydrocarbon type analysis: <sup>2/</sup>	
Paraffins and naphthenes.....	42
Olefins.....	51
Aromatics (incl. S. and N. compounds).....	7
A.S.T.M. distillation, °F. (corr. to 760 mm. Hg.)	
Initial boiling point.....	135
Percent recovered:	
5.....	163
10.....	173
20.....	186
30.....	199
40.....	211
50.....	223
60.....	238
70.....	257
80.....	281
90.....	317
95.....	347
End point.....	392
Recovery, percent.....	98.3
Residue, percent.....	.8
Loss, percent.....	.9
Reid vapor pressure, p.s.i. at 100° F. ....	4.0
Color, Saybolt.....	Below -16
Doctor test.....	Positive
Corrosion, copper strip, 3 hours at 122° F. ....	Negative
A.S.T.M. gum, mg./100 ml. ....	6
Copper dish gum, mg./100 ml. ....	21
A.S.T.M. induction period, minutes at 212° F. ....	427
Octane rating, A.S.T.M. motor method:	
Clear.....	56.1
+ 1 cc. T.E.L./gal. ....	61.0
+ 3 cc. T.E.L./gal. ....	65.5
+ 5 cc. T.E.L./gal. ....	69.8

<sup>1/</sup> Analysis made at Petroleum and Oil-Shale Experiment Station, Laramie, Wyo.

<sup>2/</sup> Analysis made on naphtha free of tar bases and tar acids.

TABLE 5. - Properties of untreated and treated pressure distillate fractions from Glen Davis, N.S.W., Australia, shale oil<sup>1/</sup>

Properties	Untreated pressure dist.	Acid treated heavy naphtha	Caustic treated light naphtha
Specific gravity, 60°/60° F. ....	0.738	0.765	0.709
Degrees A.P.I. ....	60.2	53.5	68.1
Tar bases, volume percent.....	1.0	.4	.5
Tar acids, volume percent.....	1.3	.3	.5
Sulfur, weight percent.....	.23	.31	.11
Nitrogen, weight percent.....	.13	.10	.10
Hydrocarbon type analysis: <sup>2/</sup>			
Paraffins and naphthenes.....	43	43	44
Olefins.....	46	40	52
Aromatics (incl. S. and N. compounds)..	11	17	4
A.S.T.M. distillation, °F. (corr. to 760 mm. Hg.)			
Initial boiling point.....	112	132	117
Percent recovered:			
5.....	137	182	133
10.....	151	198	139
20.....	172	219	148
30.....	193	239	156
40.....	213	257	166
50.....	235	273	180
60.....	254	288	193
70.....	275	304	205
80.....	297	324	223
90.....	323	352	236
95.....	343	398	268
End point.....	371	3/399	296
Recovery, percent.....	98.0	95.1	98.0
Residue, percent.....	1.1	3.2	.6
Loss, percent.....	.9	1.7	1.4
Reid vapor pressure, p.s.i. at 100° F...	6.0	2.5	6.9
Color, Saybolt.....	below -16	below -16	below -16
Doctor test.....	positive	positive	positive
Corrosion, copper strip, 3 hours at 122° F. ....	negative	negative	negative
A.S.T.M. gum, mg./100 ml. ....	20	561	22
Copper dish gum, mg./100 ml. ....	94	3/2,010	23
A.S.T.M. induction period, minutes at 212° F.	1,455+	1,575+	114
Octane rating, A.S.T.M. motor method:			
Clear.....	62.2	55.0	68.2
+ 1 cc. T.E.L./gal. ....	66.8	56.0	74.0
+ 3 cc. T.E.L./gal. ....	71.7	59.3	79.3
+ 5 cc. T.E.L./gal. ....	74.4	62.6	82.8
Octane rating, A.S.T.M. research method:			
Clear.....	-	67.2	77.9
Sensitivity (RM octane No. - MM octane No.).....	-	12.2	9.7

<sup>1/</sup> Analysis made at Petroleum and Oil-Shale Experiment Station, Laramie, Wyo.

<sup>2/</sup> Analysis made on naphtha free of tar acids and tar bases.

<sup>3/</sup> Sample contained heavy ends that would not evaporate.

TABLE 6. - Finished motor fuels from shale oil  
from Glen Davis, N.S.W., Australia

Properties	Sample 1 <sup>1/</sup>	Sample 2 <sup>2/</sup>	Sample 3 <sup>2/</sup>
Specific gravity, 60°/60°F. ....	0.741	0.732	0.728
Degrees A.P.I. ....	59.5	61.8	62.9
Tar bases, volume percent.....	.2	-	-
Tar acids, volume percent.....	.4	-	-
Sulfur, weight percent.....	.16	.18	.18
Nitrogen, weight percent.....	.09	-	-
Hydrocarbon type analysis: <sup>3/</sup>			
Paraffins and naphthenes.....	38	-	-
Olefins.....	47	-	-
Aromatics (incl. S. and N. compounds).....	15	-	-
A.S.T.M. distillation, °F. (corr. to 760 mm. Hg.):			
Initial boiling point.....	108	92	91
Percent recovered:			
5.....	134	124	121
10.....	148	146	134
20.....	178	170	160
30.....	210	190	182
40.....	237	210	204
50.....	259	230	223
60.....	280	250	241
70.....	299	274	263
80.....	319	300	287
90.....	349	328	316
95.....	375	346	343
End point.....	398	366	380
Recovery, percent.....	97.0	97.0	98.0
Residue, percent.....	0.6	1.5	1.0
Loss, percent.....	2.4	1.5	1.0
Reid vapor pressure, p.s.i. at 100° F. ....	7.3	7.1	7.3
Color, Saybolt.....	below -16	-	-
Doctor test.....	negative	negative	negative
Corrosion, copper strip, 3 hours at 122° F. ..	negative	negative	negative
A.S.T.M. gum, mg./100 ml. ....	2	.9	.6
Copper dish gum, mg./100 ml. ....	4	3	1
A.S.T.M. induction period, minutes at 212° F..	1,380+	-	-
Octane rating, A.S.T.M. motor method:			
Clear.....	61.1	-	-
+ 1 cc. T.E.L./gal. ....	66.6	69.1 (1.15 cc. TEL/gal.)	69.9 (>1.25 cc. TEL/gal.)
+ 3 cc. T.E.L./gal. ....	71.4	-	-
+ 5 cc. T.E.L./gal. ....	74.4	-	-
Octane rating, A.S.T.M. research method:			
Clear.....	68.2	-	-
+ 1 cc. T.E.L./gal. ....	74.2	-	-
+ 3 cc. T.E.L./gal. ....	79.2	-	-

<sup>1/</sup> Analysis made at Petroleum and Oil-Shale Experiment Station, Laramie, Wyo.

<sup>2/</sup> Analysis made at National Oil Proprietary, Ltd., laboratories, Glen Davis, N.S.W.

<sup>3/</sup> Analysis made on tar acid- and tar base-free sample.

## Finished Motor Fuel

The acid-treated heavy naphtha and polymer, after fractionation, are blended with the caustic-treated light naphtha, then doctor-treated to produce the finished motor fuel. Tetraethyllead is added to the motor fuel to raise its octane number to the 69-71 range (A.S.T.M. motor method). Table 6 shows the properties of three samples of motor fuel produced at the Glen Davis plant at different periods. Samples 2 and 3 were stock-tank samples taken in September and August 1947, respectively. The analyses of these two samples were made in the Glen Davis plant laboratories. Although the analyses show some small differences in properties, the overall quality of the product was quite consistent and met specification requirements in Australia for regular-grade motor fuel.

## RESEARCH AND ANALYSIS OF AUSTRALIAN OIL SHALES AND SHALE OIL

### Oil-Shale Research

During the last decade, several Australian investigators have made extensive studies of the origin, chemical and physical character, thermal properties, and constitution of Australian oil shales and their products. A brief review of the results of their investigations of Australian oil shales, together with some comparative analyses made in the Bureau laboratories, follows:

#### Origin of Australian Oil Shales (Torbanites)

As early as 1892, Bertrand, and Renault<sup>29/</sup> stated that the "yellow bodies" in Australian torbanite were the remains of algae, and later papers gave further proof of their contention. Dulhunty made careful microscopic studies of the physical and optical properties<sup>30/</sup> of Australian torbanites, their classification relative to other carbonaceous materials,<sup>31/</sup> and the effect of heat<sup>32/</sup> and solvents<sup>33/</sup> upon torbanites. Dulhunty, in discussing the classification of torbanites, states:

Thin sections of torbanite show a definite structure consisting of translucent organic bodies separated by films of opaque matrix. The most abundant of the organic bodies are pale yellow in color, and their substance has been referred to as "gelosite,"<sup>34/</sup> while others, orange-yellow tones, are described as consisting of "retinosite."<sup>34/</sup> These bodies are the oil-producing constituents of torbanite, frequently occupying as much as 90 percent of its volume. The two kinds of bodies, occurring side by side, are similar in all properties except refractive index, specific gravity, and color. This suggests that their difference is biological rather than the result of varying conditions of preservation. The matrix, termed "matrosite,"<sup>34/</sup> is homogeneous and opaque consisting of finely divided mineral matter with a little free carbon. Small fragments of dark brownish-red material, visible in the matrosite under high-power magnification (750 to 1,000 diameters), is referred to as "humosite,"<sup>34/</sup> and occurs in very small quantities - less than 1 percent of the total volume.

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- <sup>29/</sup> Bertrand, C. E., and Renault, B., Bull., Soc. Hist. Nat., vol. 5, 1892, p. 159.  
<sup>30/</sup> Dulhunty, J. A., The Measurement of Some Physical and Optical Properties of the New South Wales Torbanites: Proc., Linnean Soc. N.S.W., vol. 66, pts. 3-4, 1941.  
<sup>31/</sup> Dulhunty, J. A., Classification of Torbanites and Relations to Associated Carbonaceous Sediments in New South Wales: Proc., Linnean Soc. N.S.W., vol. 68, pts. 5-6, 1943.  
<sup>32/</sup> Dulhunty, J. A., The Physical Effect of Heat on Torbanites of New South Wales: Proc., Linnean Soc. N.S.W., vol. 66, pts. 5-6, 1941, pp. 335-39.  
<sup>33/</sup> Dulhunty, J. A., The Action of Solvents on Torbanite and the Nature of Extracted Products: Jour. and Proc. Royal Soc. N.S.W., vol. 76, 1943, pp. 268-274.  
<sup>34/</sup> The terms gelosite, retinosite, matrosite, and humosite apparently were coined by Dulhunty.

It is concluded that the original organic debris of torbanite consisted, essentially, of the non-vascular substance of an algae which flourished and accumulated under specialized swamp conditions.

The report points out the greatly different vascular-plant origin of cannel and other types of coals. It also discusses the influence of environment on accumulation, properties in relation to constitution and original organic debris, and other correlations and interrelations.

Dulhunty<sup>35/</sup> and Rogers and Cane<sup>36/</sup> have pointed out the similarity of formation of New South Wales torbanites to the growth and decay of algae observed to be occurring in various isolated localities such as Lake Balkash in Siberia, in the vicinity of Albany, Western Australia and in the Coorong district of South Australia, where a material called coorongite has been found floating on the surface of shallow lakes or deposited in the beds of dried-up lagoons. Coorongite when fresh resembles para rubber and on decomposition produces a large amount of petroleum-like oil. The oil, whose yield may exceed 120 gallons per ton, is similar to the oil obtained from some shales, such as the tertiary oil shales of Queensland.

Cane<sup>37/</sup> summarized the theory of torbanite origin as follows:

Torbanite originated in a specific type of colonial algae which flourished during the Permian age. Along the western margin of the Upper Coal Measures existed a series of lakes or lagoons in which algae flourished under excellent conditions but with more or less seasonal changes. Silt, clay, and other finely-divided matter containing small amounts of humus were carried down by streams into these lakes and formed a bed for the growth of the algae. After the death of the algal colonies, possibly caused by seasonal changes, the algae sank to the bottom of the water to produce a layer of sapropel. Owing to climatic changes, the lakes dried up and the algae, together with inorganic matter, caked together to form a thin layer. This cycle was repeated until an appreciable deposit was formed consisting of a layer of material of laminated structure. Separating the laminated layers were bands composed almost entirely of inorganic matter of vascular plant sapropel. Under the conditions of deposition, presumably the sapropel would be preferentially decomposed by bacterial action, and such action would cause break-down of carbohydrates and protein matter to water-soluble compounds and, to some extent, would hydrolyze the fatty matter to free acids.

Following the deposition stage, the decaying debris became covered by sediment and then, over geological time and with the pressure of overlying strata, the soft material polymerized to a hard rock. The subsided strata were brought to the surface in late Tertiary elevations and the fissures in the country exposed the torbanite as it is found today.

There is little evidence that other than plant material took part in the formation of torbanite and the lenticular shape of the deposits and their laminated structure both suggest that the plant life was of an aquatic form. The plant matter included minor amounts of spores and remains of higher plants in addition to the algae.

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<sup>35/</sup> Dulhunty, J. A., Origin of the New South Wales Torbanites: Proc., Linnean Soc. N.S.W., vol. 69, pts. 1-2, 1944.

<sup>36/</sup> Rogers, L. J., and Cane, R. F., Some Modern Trends in Oil-Shale Technology: Chem. Inst. Jour. and Proc., vol. 12, 1945, pp. 159-71. (Australian.)

<sup>37/</sup> Cane, R. F., The Chemical Constitution of Torbanite Kerogen: Jour. Soc. Chem. Inst., vol. 65, No. 12, Dec. 1946, pp. 412-14.

## Chemical Constitution of Torbanite Kerogen

Cane<sup>37/</sup> developed a hypothesis to explain the chemical structure of kerogen (the organic matter in oil shale) which accords with the foregoing theory for the formation of torbanite and is consistent with chemical activity of known compounds of structures similar to that assumed for kerogen. He suggests that the degradation of the fatty matter in the algae produces free acids that are highly unsaturated. The free acids, which are assumed to be similar to elaeostearic acid, polymerize to form a tough, semisolid, low-degree polymer. After the first stage of polymerization, the rubbery mass became covered with an overburden of rocks, and further polymerization took place until a rigid high polymer was formed. The mechanism of this type of polymerization has been proved for such types of highly unsaturated acids. The products of the thermal decomposition of polymers of this type have been shown to contain aromatic substances as well as long-chain paraffins and with decarboxylation the product was a viscous liquid resembling paraffinic lubricating oils. The fatty-acid theory of kerogen formation is therefore consistent with the presence of aromatic hydrocarbons as well as wax in crude shale oil.

Based upon theories concerning the origin of petroleum, there is also a possibility that kerogen may be derived, in part, from other portions of plants, particularly from carbohydrates. Brooks<sup>38/</sup> believed that fatty oils were the most likely source of petroleum but cellulose and protein may also contribute. Berl<sup>39/</sup> reported that cellulose and carbohydrates in the presence of alkalies or minerals as limestone, dolomite, magnesite, and zeolite are converted to a protoproduct which, in turn, yields asphalt and petroleum.

Rogers and Cane<sup>40/</sup> state, "Preliminary examination of torbanite by X-ray diffraction technique indicates that kerogen is an extensively polymerized product with no marked orientation of structure. The thickness of the crystallites as determined by an approximate form of the Scherrer equation is about  $16\text{\AA}$ ."

### Constituents of Torbanites

The inorganic constituents of Australian torbanites vary considerably with the locality of the deposit. Table 7 gives the results of oxide analyses of ashes from five different torbanites as reported by Cane.<sup>41/</sup>

TABLE 7. - Analyses of torbanite ash from various localities

Composition of ash, percent	Marangaroo	Wollar	Bong Tong Mt.	Newnes	Barigan
SiO <sub>2</sub> .....	68.2	37.5	77.4	46.8	80.7
Al <sub>2</sub> O <sub>3</sub> .....	15.2	54.1	16.3	45.1	13.4
Fe <sub>2</sub> O <sub>3</sub> .....	11.5	3.9	4.0	2.9	5.1
CaO.....	1.9	1.4	1.0	2.6	.4
MgO.....	1.1	.3	2	.9	.3

<sup>38/</sup> Brooks, Benjamin T., The Origin of Petroleum in the Light of Recent Research: Calif. Oil World, vol. 42, No. 1, 1949, pp. 1-11; No. 2, 1949 pp. 3-13.

<sup>39/</sup> Berl, E., The Origin of Petroleum: Trans. Am. Inst. Min. Eng., Petrol. Div., vol. 127, 1938, pp. 99-116.

<sup>40/</sup> See footnote 36.

<sup>41/</sup> See footnote 37.

Although the elements given in the above table constitute about 98 percent of the total mineral matter, the residual 2 percent contains numerous other elements. The ash from the Marangaroo deposit was examined by means of a large Hilger quartz spectrograph and the following metals were found to be present, in addition to those given above: manganese, zinc, lead, tin, iridium, vanadium, titanium, antimony, bismuth, molybdenum, beryllium, sodium, silver, strontium, nickel, chromium, mercury, and copper.

Cane suggests the possibility of some of the metallic elements occurring as organo-metallic complexes.

Table 8 gives the mineral composition and fusion points of the shale ashes, properties of the raw shale, their organic contents, and the quantities of carbon and hydrogen in the organic matter of the main seam and top seam shales from Glen Davis and of the Coolaway shale from the Rylstone area.

Table 8. - Properties of Australian oil shales<sup>1/</sup>

	Source		
	Glen Davis, N.S.W., main seam	Glen Davis, N.S.W., top seam	Rylstone area, N.S.W., Coolaway
Ash, percent.....	44.54	63.28	4.44
Mineral composition of ash, percent:			
SiO <sub>2</sub> .....	90.72	70.93	59.59
Al <sub>2</sub> O <sub>3</sub> .....	4.84	19.20	27.14
Fe <sub>2</sub> O <sub>3</sub> .....	0.91	2.69	2.02
CaO.....	.54	1.87	2.71
MgO.....	.24	1.62	0.84
SO <sub>3</sub> .....	.19	.73	3.13
Undetermined (mostly alkalis).....	2.56	2.96	4.57
Total.....	100.00	100.00	100.00
Fusion point of shale ash, °F. ....	Above 2,570°	2,375°	-
Properties of raw shale:			
Mineral CO <sub>2</sub> , percent.....	.04	2.49	.08
Gross heating value, B.t.u./lb. ....	9,250	5,070	17,090
Specific gravity of minus 8 mesh shale at 60/60° F. ....	1.403	1.874	1.043
Total nitrogen, percent.....	.47	.71	.83
Total sulfur, percent.....	.44	.47	.49
Organic material:			
Approximate organic content, percent <sup>2/</sup>	55.42	34.23	95.48
Carbon, percent.....	46.47	27.60	81.44
Hydrogen, percent <sup>3/</sup> .....	5.75	2.40	10.46
C/H ratio.....	8.08	11.50	7.79

<sup>1/</sup> Analyses made at Petroleum and Oil-Shale Experiment Station, Laramie, Wyo.

<sup>2/</sup> Total volatile minus mineral CO<sub>2</sub>.

<sup>3/</sup> Not corrected for possible water of crystallization in mineral matter.

## Assay for Oil Yield

The modified Gray-King method of oil-shale assay is used in Australia. The Gray-King method<sup>42/</sup>,<sup>43/</sup> was developed originally for coal analysis but has been modified to make it more applicable to oil-shale assay. The method consists essentially of introducing a 30-gram sample of minus 60-mesh shale into a tube heated to 400° C. in a combustion type furnace, raising the temperature gradually to 600° C. over a period of 1 hour, then holding the temperature at 600° C. for at least an additional half hour, and collecting the oil product in a bulb-bottom U-tube receiver with the outlet arm packed in dry ice to insure complete condensation.

Because of the great differences in apparatus and temperatures used in the modified Gray-King and modified Fischer methods<sup>44/</sup> for assaying oil shales, assays were made by each method on duplicate shale samples, so that results by either method may be evaluated in terms of the other. Three Glen Davis shale samples were carefully prepared and assayed in the National Oil Proprietary laboratories. Sealed duplicates of these samples were shipped to the Bureau of Mines at Laramie, Wyo., where Fischer assays were made. Table 9 gives the results of these comparative tests.

TABLE 9. - Comparison of assay results by National Oil Proprietary, Ltd.,<sup>1/</sup>  
and by the Bureau of Mines<sup>2/</sup>

Sample and laboratory	Yield of products						Specific gravity of oil, 60°/60°F.
	Weight percent				Oil		
	Oil	Water	Spent shale	Gas + loss	U.S. gal./ton	Imp. gal./ton	
<b>Secondary seam:</b>							
1. Nat'l. Oil Proprietary..	8.5	2.1	84.3	5.1	23.4	21.8	0.870
2. Bureau of Mines.....	8.6	2.9	86.4	2.1	22.8	21.3	.906
Difference (1-2).....	-0.1	-0.8	-2.1	+3.0	+0.6	+0.5	-0.036
<b>Daily shale:</b>							
1. Nat'l. Oil Proprietary..	20.1	1.1	73.0	5.8	55.2	51.5	0.873
2. Bureau of Mines.....	21.1	2.1	73.2	3.6	56.8	53.0	.888
Difference (1-2).....	-1.0	-1.0	-0.2	+2.2	-1.6	-1.5	-0.015
<b>Main seam:</b>							
1. Nat'l. Oil Proprietary..	52.2	0.7	41.1	6.0	150.3	140.2	0.834
2. Bureau of Mines.....	53.1	.5	39.8	6.6	143.5	133.8	.887
Difference (1-2).....	-0.9	+0.2	+1.3	-0.6	+6.8	+6.4	-0.053

<sup>1/</sup> Modified Gray-King method.

<sup>2/</sup> Modified Fischer method.

The yields of oil, on a weight basis, agreed within 1 percent, the Fischer assay giving the higher yield. However, the Gray-King method produces a lower-gravity oil,

<sup>42/</sup> Gray and King, Fuel Research Board (Great Britain), Tech. Paper 1, 1921.

<sup>43/</sup> Davis, J. D, and Galloway, A. E., Low-Temperature Carbonization of Lignites and Sub-Bituminous Coals: Ind. Eng. Chem., vol. 20, No. 6, June 1928, pp. 612-17.

<sup>44/</sup> Stanfield, K. E., and Frost, I. C., Method of Assaying Oil Shale by a Modified Fischer Retort: Bureau of Mines Rept. of Investigations 3977, 1946, 11 pp., 5 figs.



so when yields are converted to a volume basis in terms of gallons per ton, the Gray-King values are usually somewhat higher, and comparative yields are more erratic. The principal differences in the two methods seem to be the greater degree of decomposition of the shale and more efficient recovery of light naphtha by the Gray-King method, which results in an oil of lower gravity and a corresponding difference in the oil yields by volume.

The three additional samples of Australian shales, for which ultimate and mineral analyses are given in table 8, were assayed by the Bureau of Mines with the modified Fischer method, and analyses were made of the gases produced by the assaying procedure. The results of assay and gas analyses of these three samples are shown in table 10.

TABLE 10. - Assays of Australian oil shales and analyses of their assay gases<sup>1/</sup>

Sample source Sample No.	Glen Davis (main seam) 591	Glen Davis (top seam) 592	Rylstone (Coolaway) 593
Yield of products:			
Oil, weight percent.....	40.3	8.4	74.9
Water, weight percent.....	.5	2.2	.5
Spent shale, weight percent.....	53.6	86.9	15.2
Gas + loss, weight percent.....	5.6	2.5	9.4
Oil, gal./ton.....	108.0	21.2	200.0
Water, gal./ton.....	1.2	5.4	1.2
Sp. gr., oil.....	.895	.945	.899
Properties of spent shale:			
Ignition loss, percent of raw shale.....	13.3	23.2	-
Ash, percent of raw shale.....	40.3	63.7	-
Assay retort gas:			
Specific gravity (air = 1).....	1.004	0.998	0.995
Yield, cu. ft./ton.....	1,125	976	1,428
Composition <sup>2/</sup> (air-free):			
Methane.....	31.1	41.7	24.9
Ethane.....	14.6	11.2	12.9
Propane.....	7.1	2.7	6.9
n-Butane.....	3.5	1.0	3.9
Isobutane.....	.1	.5	.1
n-Pentane.....	1.8	.5	2.2
Isopentane.....	.4	.4	.1
Hexanes.....	.8	.2	.6
Carbon dioxide.....	3.5	10.9	6.0
Carbon monoxide.....	3.3	3.4	10.0
Nitrogen.....	7.7	6.7	6.4
Hydrogen.....	6.1	12.8	5.3
Ethylene.....	4.7	2.3	4.9
Propylene.....	6.8	2.3	7.0
Butene-1 } .....	3.7	1.5	4.3
Butene-2 } .....			
Isobutene } .....			
Pentenes.....	2.8	1.1	3.2
Hexenes.....	1.2	.4	1.1
Hydrogen sulfide.....	.9	.3	.2
Argon.....	-	.1	-

<sup>1/</sup> Analyses made at Petroleum and Oil-Shale Experiment Station, Laramie, Wyo.

<sup>2/</sup> Analysis made by mass spectrometer.

The Coolaway sample was representative of an exceptionally rich shale from the Coolaway area near Rylstone, New South Wales, about 30 miles northwest of Glen Davis. Small deposits of torbanite have been found that are richer than the Coolaway samples. A small deposit of what is believed to be the richest oil shale in the world was discovered in the Marangaroo area southwest of Glen Davis. Cane<sup>45/</sup> described this material as yielding 237 Imperial gallons of oil per long ton and as having the following proximate analysis:

	<u>Percent</u>
Moisture.....	0.2
Volatile matter.....	93.1
Fixed carbon.....	4.9
Ash.....	1.8

#### The Mechanism of Decomposition of Torbanite Kerogen

In 1942, Cane<sup>46/</sup> described the conversion of the organic matter in torbanites as taking place in two steps or stages: (1) "depolymerization" of kerogen to a heavy bitumen and (2) cracking of the bitumen to shale oil. In 1945, Rogers and Cane<sup>47/</sup> showed that the conversion of kerogen to so-called "bitumen" was at least a two-stage process. Their work showed that upon heating the torbanite to progressively higher temperatures, it expands and darkens presumably by oxidation. When the temperature reaches about 250° C., gases consisting mainly of hydrogen sulfide, carbon dioxide, and water vapor, but virtually free from hydrocarbons, are evolved. Whether the source of the gases is organic or inorganic is not proven, but the evidence indicated they were formed mainly by dehydrogenation of the kerogen. At this stage, the torbanite showed a marked change in character, becoming soft and somewhat resilient, but remained insoluble in organic solvents. The term "rubberoid" was suggested for the torbanite product in this stage of decomposition. On further heating, hydrocarbon gases are evolved at a temperature of about 320° C., and the "rubberoid" is converted into a secondary product, termed "bitumen" in the sense that it is a high-boiling, viscous material, but it contains little or no asphaltic material. The bitumen is completely soluble in either aromatic or aliphatic solvents. The formation of oil from the bitumen is considered by the investigators to be a normal cracking reaction.

In 1948, Cane<sup>48/</sup> discussed the subject in more detail and gave more analytical evidence to support further the earlier theory. Assuming the initial oxidation of kerogen to be the first stage of the decomposition process, kerogen is converted to oil in four stages, i.e.: (1) kerogen to oxidized kerogen, (2) to rubberoid, (3) to bitumen, and (4) to oil. Based on the change in carbon-hydrogen ratio, Cane believes the breakdown of kerogen to bitumen is not "depolymerization" but gentle cracking; thus, the whole reaction from kerogen to oil is one of pyrolysis or thermal cracking. He summarizes the complete conversion reaction as follows:

In the original kerogen, the molecule consists of many units, each containing one or more cyclic nuclei possessing long side chains. The occurrence of paraffin wax in the crude oil leaves no doubt that long chain paraffin structures are present, as such, in the kerogen molecule.

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<sup>45/</sup> Cane, R. F., Rich Torbanite from Marangaroo: Australian Jour. Sci., vol. 5, No. 5, 1943, p. 156.

<sup>46/</sup> Cane, R. F., Some Thermochemical Properties of the Torbanite of the Glen Davis Deposit: Jour. & Proc., Royal Soc. N.S.W., vol. 76, 1943, pp. 190-202.

<sup>47/</sup> See footnote 36.

<sup>48/</sup> Cane, R. F., The Chemistry of the Pyrolysis of Torbanite: Australian Chem. Inst. Jour. & Proc., Feb. 1948, pp. 62-8.

Furthermore, as the number of carbon atoms in the wax may reach twenty-six per molecule, it must be concluded that the kerogen molecule contains an equal or greater number of carbon atoms in straight-chain structures. These side chains are, in the main, saturated, but ethylene linkages are found in certain members. In addition to the long side chains, each benzene nucleus has a few short chains.

As the kerogen is heated slight oxidation takes place, followed by the initial decomposition, causing loss of sulfur, hydrogen, and oxygen, concurrently with the disappearance of some cross-linkages. The result of these reactions is a general softening of the kerogen, accompanied by evolution of hydrogen sulfide and carbon dioxide.

As the reaction proceeds, cross-linkages are progressively ruptured until a stage is reached where the macro-molecule no longer exists, and the structure is essentially elastomeric.

The few remaining cross-linkages disappear in the next stage, and at the same time some side-chains have received sufficient activation to break away from the parent ring. This stage would correspond to a semi-decomposed state in which small fragments of the original structure co-exist with long-chain paraffins from the side chains, olefins from the cracking of the paraffins, and cyclic structures from condensation, or ring closure, etc. This heterogeneous mixture constitutes the "bitumen" discussed earlier.

From this stage onward, no general plan is followed. Cracking would occur in the normal manner and those principles found to apply to the cracking of flow oil (petroleum) would be equally tenable from the bitumen stage until the decomposition is complete; the final products being oil, gas, and carbonaceous residue.

#### Heat Factors Involved in the Conversion of Australian Oil Shales

The different heat factors involved in the conversion of Australian oil shales to oil, gas, and coke have been determined or calculated, and the results have been reported largely by Cane.<sup>49/</sup> The following summary of experimental work and values was taken from Cane's report:

The specific heat of the mineral portion of the shale was calculated from the chemical constituents of the ash of a representative shale sample from Glen Davis. This calculation gave a value of 0.19 for the specific heat of the mineral constituents. The specific heat of the organic constituent was calculated to be 0.38, based on the percentage composition of the kerogen and the atomic heats of the constituent atoms. Thus a shale with 50 percent mineral matter would have a theoretical specific heat of 0.28.

The method of mixtures was used to determine the specific heats of four shales having a broad range of ash content. The determined values are compared with values obtained by the foregoing "calculation" method in the following table:

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<sup>49/</sup> See footnote 46.

Ash, percent of original shale	Specific heat, observed	Specific heat, calculated	Temperature range, °C.
22.2.....	0.318	0.336	20-50
32.3.....	.301	.316	30-80
56.4.....	.278	.271	20-50
86.2.....	.242	.214	20-50

The above values for specific heat are in reasonable agreement with those determined for Colorado shales by Shaw,<sup>50/</sup> considering the differences in the mineral constituents of the two types of shales.

The quantity of heat required for the conversion reaction of shale to oil appears to be an elusive value, as about as many values are reported for individual shales throughout the world as there were investigators and methods. The values reported for Australian shales are no exception.

Cane<sup>51/</sup> performed some qualitative experiments in which two silica tubes, one containing shale and the other dehydrated fullers earth, were heated simultaneously in the same furnace, and the differential temperature between the shale and fullers earth was plotted over the temperature range 300° to 550° C. These experiments indicated that the reaction was endothermic to about 365° C., exothermic from 365° to 465° C., again endothermic from 465° to 530° C., and exothermic above 530° C. McKee and Goodwin<sup>52/</sup> showed similar relationships for oil shales and coal. It seems quite probable that several heat-counterbalancing reactions occur simultaneously, thus making the problem of obtaining a net heat of reaction a difficult one.

Rogers and Cane<sup>53/</sup> reported the heat of reaction of torbanite from Rylstone, N.S.W., ranged from 784 to 1,180 calories per gram of oil and gas formed when determined by a method similar to that used by McKee and Lyder,<sup>54/</sup> which is about twice the value reported by McKee and Lyder. Rogers and Cane, in the same article, reported a value of 280 calories per gram of oil and gas formed for the heat of reaction calculated from the operation of a 50-ton-a-day pilot retort when retorting Baerami, N.S.W., shale. Cane<sup>55/</sup> calculated an approximate value for the heat of reaction by subtracting the heat evolved when the total products were oxidized completely from that required for the complete oxidation of an equivalent quantity of raw shale. He obtained a value of 45 calories per gram of shale, or 86 calories per gram of oil produced.

Cane gave the heat-of-combustion value for kerogen as 10,680 gram calories obtained by extrapolating the calorific values of several shales. A theoretical value of 10,003 gram calories was obtained by calculation from the elemental composition of kerogen. Similarly, the mean value of the heat of combustion for Australian shale oil was determined as 10,872 gram calories, and the theoretical value as 10,737 gram calories from its ultimate composition.

<sup>50/</sup> Shaw, R. J., Specific Heat of Colorado Oil Shales: Bureau of Mines Rept. of Investigations 4151, 1947, 9 pp.

<sup>51/</sup> See footnote 46.

<sup>52/</sup> McKee, Ralph H., Shale Oil: A.C.S. Monograph No. 25, Chemical Catalog Co., New York, 1925, pp. 76-79.

<sup>53/</sup> See footnote 36.

<sup>54/</sup> McKee, Ralph H., and Lyder, E. E., Thermal Decomposition of Shales, Heats of Reaction: Jour., Ind. Eng. Chem., vol. 13, 1921, pp. 678-684.

<sup>55/</sup> See footnote 46.

A comparison of the rates of decomposition of Australian oil shale at 400° and 450° C. for several particle sizes was made by Cane<sup>56/</sup> with a unique apparatus, based on the torsion-balance principle, to measure continuously the loss in weight of the sample while it was being heated. The results showed the rate of decomposition to be a function of particle size, with greatly increased rates at the higher temperature.

#### Laboratory and Pilot-Plant Studies of Oil-Shale Conversion Processes

In 1942<sup>57/</sup> and 1943<sup>58/</sup> Dulhunty reported the results of heating torbanite in the presence of a solvent to conversion temperatures under pressure. Coal-tar naphtha (150°-220° C.), petroleum kerosine (190°-210° C.), and torbanite crude-oil distillate (230°-260° C.) were used as solvents. Some experiments were carried out in which the torbanite was first preheated and then extracted with solvents, but in most of the experiments the torbanite and solvent were mixed and heated to the desired temperature, and a pressure was maintained on the system to maintain the solvent in the liquid phase.

In one experiment, the shale-solvent mixture was heated first to 375° C., then successively to higher temperatures and pressures after each of seven cycles. Gas and oils boiling to 230° C. were removed after each cycle. Other experiments were made in which naphtha vapors were removed continuously as the mixture was heated gradually from about 350° to 400° C. The final experiment consisted of heating the mixture to 390° C. and holding that temperature for 6 hours without removing products.

Cycle heating gave a high yield of 84 percent of the total organic matter but low yields of gas and naphtha, the product being a relatively high-boiling oil. By a continuous removal of products, the yield based on total organic matter was somewhat lower but the yield of gas and naphtha was higher than by cycle heating. By continued heating without removal of products, a much lower yield based on total organic was obtained, but the product was approximately 50 percent naphtha boiling below 180°C.

Additional experimental work was done on the "solution-cracking" type of processing on a laboratory and pilot-plant scale at the National Oil Proprietary Laboratories at Glen Davis, but the results of this later work have not been published.

Early in World War II consideration was given to the possible production of marine fuel from Australian shales because of the long, dangerous tanker voyage from the United States to Australia and the South Pacific and the necessity for tankers to retain enough fuel for the return trip. Early in 1942, the Standard Oil Development Co. ran pilot-plant tests<sup>59/</sup> to determine the feasibility of applying the fluidized-solids technique to retorting Australian oil shales for the production of heavy fuels. There was no great need for recovering the low-boiling products, and much steel could be saved by reducing the condensing equipment to a minimum; consequently, no attempt was made to produce or recover light products.

The experimental retort consisted of a single chamber in which shale of 1/4-inch mesh and finer was fluidized by means of air and air and steam. Temperatures ranging

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<sup>56/</sup> See footnote 46.

<sup>57/</sup> See footnote 33.

<sup>58/</sup> Dulhunty, J. A., Preliminary Notes on Solution-Cracking Treatment of Torbanite: Jour. and Proc. Royal Soc. N.S.W., vol. 77, 1943, pp. 24-32.

<sup>59/</sup> Blanding, F. H., and Roethell, B. E., Retorting Oil Shale by the Fluidized Solids Technique: Oil & Gas Jour., vol. 45, No. 41, Feb. 15, 1947, pp. 84, 86-8, 96.

from 850° F. to 1,000° F. were produced by burning with air the carbon left on the spent shale and possibly some distilled oil vapors. Residence times ranged between 8 and 20 minutes. Spent shale fines and dust were removed in a filter section, and the vapors were partly condensed at 300° F. to separate a heavy oil, then the light vapors and water were condensed at 90° F.

Based on the results of the pilot-plant study, a preliminary process design was prepared. The report discusses the effect of varying operating conditions, gives product analyses, and suggests possible alternative methods of operation.

Rogers and Cane<sup>60/</sup> pointed out a number of basic factors that should be met in commercial retorting operations. They discuss several different retorting processes with reference to their ability to give the desired results, particularly their applicability to Australian shales.

#### Shale-Oil Research

In addition to the research work on the character of oil shales and the refining characteristics of shale oils previously discussed, considerable basic research has been done on the chemical character of shale oil, with particular reference to the sulfur and nitrogen compounds and methods for their detection and analysis.

Mapstone, the present chief chemist of the National Oil Proprietary laboratories, has published a number of reports covering this work. He developed a nitroprusside test<sup>61/</sup> for the detection of mercaptans in kerosine and cresylic acids that is at least as sensitive and more readily discernible at low mercaptan concentrations than the doctor test. The replacement of the customary water washing after sweetening with a caustic soda washing<sup>62/</sup> proved to be a satisfactory method for removing traces of lead mercaptide from the gasoline without the addition of free sulfur. The method of Borgstrom and Reid<sup>63/</sup> was modified to render it applicable to the determination of hydrogen sulfide and mercaptans in cresylic acid samples.<sup>64/</sup> Mapstone<sup>65/</sup> made a study of the effectiveness of a number of adsorbents for the removal of sulfur compounds from gasoline and kerosine distillates. Silica gel and alumina were found the most effective; however, even these adsorbents removed sulfur for a short time only, and subsequent desorption of sulfur compounds increased the sulfur content of the filtrate above that of the original sample. Conversion of a portion of the mercaptans to other types of sulfur compounds was indicated, as the filtrate and oil recovered from the adsorbent did not yield the original quantity of mercaptans. Mapstone<sup>66/</sup> studied the sensitivity of various tests for the detection of elemental sulfur in gasoline and found that a modification of the Sommer test was about five times as sensitive as the customary inverted doctor test and less sensitive but more convenient for plant control than the mercury test.

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<sup>60/</sup> See footnote 36.

<sup>61/</sup> Mapstone, G. E., The Detection of Mercaptans: Australian Chem. Inst. Jour. and Proc., vol. 13, July 1946, pp. 269-74.

<sup>62/</sup> Mapstone, G. E., Some Notes on the Doctor Treatment of Gasoline: Australian Chem. Inst. Jour. and Proc., vol. 13, April 1946, pp. 156-60.

<sup>63/</sup> See footnote 28.

<sup>64/</sup> Mapstone, G. E., The Determination of Hydrogen Sulfide and Mercaptans in Cresylic Acid: Australian Chem. Inst. Jour. and Proc., vol. 13, Oct. 1946, pp. 373-77.

<sup>65/</sup> Mapstone, G. E., Some Notes on the Desulfurization of Gasoline by Adsorption: Australian Chem. Inst. Jour. & Proc., vol. 14, Feb. 1947, pp. 61-7.

<sup>66/</sup> Mapstone, G. E., Detection of Elemental Sulfur in Gasoline by the Sommer Test: Ind. Eng. Chem. (anal. ed.), vol. 18, Aug. 15, 1946, pp. 498-99.

Mapstone and his associates have done a large amount of work, at present unpublished, on the nitrogen compounds in oil shales and shale oils. These studies range from improvement of the Kjeldahl method for nitrogen determination to the use of tar bases as insecticides and germicides and include such other subjects as the source of nitrogen compounds in kerogen, nonbasic nitrogen compounds in shale-oil distillates, the increase in tar base content of fractions by thermal cracking, and a review of the nitrogen-base compounds found in shale oils throughout the world.

A number of additional publications by Mapstone<sup>67/</sup> and his associates have resulted from the solution of various operating, control, and analytical problems at the National Oil Proprietary oil-shale plant at Glen Davis.

#### Glen Davis Laboratories

The laboratories of the National Oil Proprietary plant at Glen Davis consist of a main laboratory, which is a one-story building; a second smaller one-story building housing the gas-analysis laboratory, crushing room, lavatory, and showers; and pilot-plant equipment, which is not housed. The main building contains the chief chemist's office, library, CFR test engine room, sample room, storage room, and two laboratory rooms - one large and one small - in which most of the control testing, analytical work, and research are done.

The laboratory facilities and equipment are normal to those found in small petroleum refineries, together with additional equipment for analyzing and testing raw materials and products peculiar to the oil-shale industry, such as assay, mineral analysis, nitrogen determination, etc. Unfortunately, owing to lack of funds, the most recently developed physical instruments, which would be of great aid to their research and analytical work, are lacking. The laboratory work is primarily control testing and the solution of plant-operating problems, research work being done by the same personnel as time becomes available from the required testing work.

The laboratory personnel consists of the chief chemist, two "qualified" (graduate) chemists, and 22 "unqualified" (undergraduate) chemists. The "unqualified" chemists have met college entrance requirements and, through a cooperative agreement with the Sydney Technical College at Sydney, the "qualified" chemical staff at the plant teach technical subjects at the college level, for which the students receive college credit.

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- <sup>67/</sup> Mapstone, G. E., The Regeneration of Acid CuCl solution; Australian Chem. Inst. Jour. & Proc., vol. 10, 1943, 267-8.
- Modified St. Claire Deville Method for Estimation of Naphtha in Shale Gas. Australian Chem. Inst. Jour. & Proc., vol. 11, 1944, pp. 191-6.
- Nomograph for Absorption Factor Equation. Ind. Eng. Chem., vol. 37, Dec. 1945, pp. 1222-3.
- The Spontaneous Evaporation of a Two-Component Mixture. Australian Chem. Inst. Jour. & Proc., vol. 13, 1946, pp. 60-4.
- Treatment of Hard-Bore Water for Industrial Purposes. Chem. Eng. Min. Rev., vol. 39, 1946, pp. 87-9.
- The Colorimetric Determination of Tar Acids in Gasoline. Australian Chem. Inst. Jour. & Proc., vol. 15, 1948, pp. 9-12.
- Vapor Pressure Nomograph for Hydrochloric Acid. Australian Chem. Inst. Jour. & Proc., vol. 14, 1947, pp. 338.
- Mapstone, G. E., and Beckmann, P., Determination of Olefins in Cracked Gases: Petr. Ref., vol. 27, No. 8, August 1948, pp. 112-16.
- Mapstone, G. E., and Provera, F. M., A Magnetic Boiler Scale: Australian Chem. Inst. Jour. & Proc., vol. 13, 1946, pp. 369-70.

Three years of credit toward a 5-year diploma course in chemical engineering may be received by the students under this arrangement. The compensation of the student chemist is raised in accordance with his technical advancement. This teaching system places a heavy burden on the technical staff, as all the teaching must be done outside of regular working hours, but it has been a considerable aid to this rather isolated location in obtaining better-quality laboratory personnel. It has served, also, to make the laboratory a training ground for refinery personnel, a most important function in a country where such training is not generally available.

#### CONCLUSIONS

Oil shales of Australia, and particularly those found in New South Wales, have been studied extensively, and much interesting information has been obtained bearing upon the origin and composition of these shales and their organic components. The oil shales of New South Wales resemble, in general characteristics, the Scottish torbanites. The Australian shales occur in small isolated deposits, which are usually lenticular and thin-bedded, causing difficult and costly mining operations.

Apparently the first oil-shale retorting in Australia was done in 1865. The only sustained commercial operation in recent years has been that of National Oil Proprietary, Ltd., at Glen Davis, N. S. W. The oil shale charged to the retorts is the richest (67 U. S. gallons per short ton) that is being charged to any oil-shale plant in the world that is operated for the manufacture of liquid fuels. However, the success of the project has been hampered by high mining costs and inability to mine enough shale to keep the retort batteries operating at full capacity, and to operate the refinery as much as half time. This reduced scale of operation also has increased the unit costs of marketable products.





